Supporting information

Iron-catalysed tandem isomerisation/hydrosilylation reaction of allylic alcohols with amines

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I. General information.

All reagents were obtained from commercial sources and used as received. All reactions were carried out with flame-dried glassware using standard Schlenk techniques under an inert atmosphere of dry argon. Dichloromethane, toluene and THF were dried over Braun MB-SPS-800 solvent purification system. Ethanol is distilled under reduced pressure and stored in the presence of molecular sieves. Technical grade petroleum ether (40-60 °C bp.) and diethylether were used for chromatography column. Analytical TLC was performed on Merck 60F254 silica gel plates (0.25 mm thickness). Column chromatography was performed on Acros Organics Ultrapure silica gel (mesh size 40-60 μm, 60 Å).

$^1$H NMR spectra were recorded in CDCl$_3$ at ambient temperature on Bruker AVANCE 300 and 400 spectrometers at 300.1, and 400.1 MHz, respectively, using the solvent as internal standard (CDCl$_3$ 7.26 ppm). $^{13}$C NMR spectra were obtained at 75 or 100 MHz and referenced to the internal solvent signals (CDCl$_3$, central peak is 77.16 ppm, C$_6$D$_6$ 128.06 ppm). Chemical shifts and coupling constants ($J$) are given in ppm and in Hz, respectively. The peak patterns are indicated as follows: (s, singlet; d, doublet; t, triplet; q, quartet; quint, quintet; m, multiplet, and br. for broad).

GC analyses were performed with GC-2014 (Shimadzu) 2010 equipped with a 30-m capillary column (Supelco, SPBTM-20, fused silica capillary column, 30 m × 0.25 mm × 0.25 mm film thickness), which was used with N$_2$/air as the vector gas. The following GC conditions were used: initial temperature 80 °C, for 2 minutes, then rate 10 °C/min. until 220 °C and 220 °C for 15 minutes.

GC-MS were measured by GCMS-QP2010S (Shimadzu) with GC-2010 equipped with a 30-m capillary column (Supelco, SLBTM-5ms, fused silica capillary column, 30 m × 0.25 mm × 0.25 mm film thickness), which was used with helium as the vector gas. The following GC-MS conditions were used: initial temperature 100 °C, for 2 minutes, then rate 10 °C/min. until 250 °C and 250 °C for 10 minutes.

HR–MS spectra and elemental analysis were carried out by the corresponding facilities at the CRMPO (Centre Régional de Mesure Physiques de l’Ouest), University Rennes 1.

Visible light irradiation experiments were performed using a 24 Watt compact fluorescent lamp.
II- General experimental procedures:

A 20 mL oven dried Schlenk tube containing a stirring bar, was charged with Fe(cod)(CO)$_3$ (3.1 mg, 0.0125 mmol) and then purged with argon/vacuum three times. Ethanol (1 mL), aniline derivative (0.25 mmol, 1 equiv.), allylic alcohol or homoallylic alcohol (1.0-1.5 equiv.), PMHS (45 μL, 3 equiv.) were added under argon. The reaction mixture was stirred in a preheated oil bath for 20 h under visible light irradiation. The reaction mixture was condensed under reduced pressure. The residue was then purified by silica gel column chromatography using a mixture of diethylether/petroleum ether as the eluent to afford the desired product.

Procedure A: allylic alcohol or homoallylic alcohol (0.375 mmol, 1.5 equiv.), heated at 50 °C.
Procedure B: allylic alcohol (0.313 mmol, 1.25 equiv.), heated at 70 °C.
Procedure C: allylic alcohol (0.25 mmol, 1.0 equiv.), heated at 70 °C.
Procedure D: allylic alcohol (0.313 mmol, 1.25 equiv.), heated at 100 °C.

III- Characterization data for the aniline derivatives

N-Ethyl-N-hexylaniline (Table 2, Entries 1, 2, 10)$^1$

\[
\begin{array}{c}
\text{N} \\
\text{\begin{tikzpicture}
\draw (0,0) -- (0.5,1) -- (1,0);
\end{tikzpicture}}
\end{array}
\]

The compound was prepared as described using the procedure A (m = 43 mg, 86% isolated yield).
$^1$H NMR (400 MHz, CDCl$_3$) δ 7.21 (t, 2H, $J = 7.9$), 6.70-6.59 (m, 3H), 3.36 (q, 2H, $J = 7.0$), 3.25 (t, 2H, $J = 7.7$), 1.59 (m, 2H), 1.33 (m, 6H), 1.15 (t, 3H, $J = 6.9$), 0.91 (t, 3H, $J = 6.5$).
$^{13}$C{$^1$H} NMR (101 MHz, CDCl$_3$) δ 148.0, 129.2, 115.2, 111.7, 50.5, 44.9, 31.8, 27.5, 26.9, 22.7, 14.0, 12.3.

N-Butyl-N-ethylaniline (Table 2, Entries 3, 9)$^1$

\[
\begin{array}{c}
\text{N} \\
\text{\begin{tikzpicture}
\draw (0,0) -- (0.5,1) -- (1,0);
\end{tikzpicture}}
\end{array}
\]

The compound was prepared as described using the procedure A (m = 36 mg, 81% isolated yield).
$^1$H NMR (400 MHz, CDCl$_3$) δ 7.21 (t, 2H, $J = 7.6$), 6.70-6.60 (m, 3H), 3.36 (q, 2H, $J = 7.1$), 3.25 (t, 2H, $J = 7.4$), 1.63 – 1.51 (m, 2H), 1.42 – 1.31 (m, 2H), 1.15 (t, 3H, $J = 7.0$), 0.96 (t, 3H, $J = 7.3$).
$^{13}$C{$^1$H} NMR (101 MHz, CDCl$_3$) δ 148.0, 129.2, 115.2, 111.8, 50.2, 44.9, 29.7, 20.4, 14.0, 12.3.
**N-Ethyl-N-isopentylaniline (Table 2, Entries 4, 11)**

![N-Ethyl-N-isopentylaniline](image)

The compound was prepared as described using the *procedure A* (m = 39 mg, 82% isolated yield).

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.20 (t, 2H, $J = 7.6$), 6.70-6.60 (m, 3H), 3.35 (q, 2H, $J = 7.1$), 3.27 (t, 2H, 7.8), 1.70 – 1.56 (m, 1H), 1.48 (m, 2H), 1.15 (t, $J = 7.0$ Hz), 0.96 (d, $J = 6.6$ Hz).

$^{13}$C$^1$H NMR (101 MHz, CDCl$_3$) $\delta$ 148.0, 129.2, 115.2, 111.8, 48.7, 44.8, 36.2, 26.4, 22.7, 12.4.

MS (EI): m/z: 191 (8, M+), 134 (100), 120 (12), 106 (28), 77 (15).

**N-Ethyl-N-isobutylaniline (Table 2, Entry 5)**

![N-Ethyl-N-isobutylaniline](image)

The compound was prepared as described using the *procedure A* (m = 42 mg, 95% isolated yield).

$^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 7.26-7.16 (m, 2H), 6.81-6.54 (m, 3H), 3.41 (q, 2H, $J = 7.2$), 3.05 (d, 2H, $J = 7.3$), 2.12-2.19 (m, 1H), 1.14 (t, 3H, $J = 7.2$), 0.95 (d, 6H, $J = 6.2$).

$^{13}$C$^1$H NMR (75 MHz, CDCl$_3$) $\delta$ 148.3, 129.1, 115.2, 112.0, 58.5, 45.8, 27.2, 20.4, 11.6.

**N-Ethyl-N-(3-phenylpropyl)aniline (Table 2, Entry 6)**

![N-Ethyl-N-(3-phenylpropyl)aniline](image)

The compound was prepared as described using the *procedure A* (m = 45 mg, 75% isolated yield).

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.30 (m, 2H), 7.24-7.16 (m, 5H), 6.68-6.10 (m, 2H), 3.37 (q, 2H, $J = 6.9$), 3.30 (t, 2H, $J = 6.5$), 2.68 (t, 2H, $J = 7.6$), 2.00-1.88 (m, 2H), 1.15 (t, 1H, $J = 6.9$).

$^{13}$C$^1$H NMR (101 MHz, CDCl$_3$) $\delta$ 148.0, 141.8, 129.1, 128.40, 128.37, 125.9, 115.5, 112.0, 50.0, 45.0, 33.4, 29.0, 12.4.

**N-Ethyl-N-(3,7,11,15-tetramethylhexadecyl)aniline (Table 2, Entry 8)**

![N-Ethyl-N-(3,7,11,15-tetramethylhexadecyl)aniline](image)

The compound was prepared as described using the *procedure A* (m = 65 mg, 65% isolated yield).

$^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 7.25-7.17 (m, 2H), 6.74-6.59 (m, 3H), 3.37 (q, 2H, $J = 7.1$), 3.20-3.19 (m, 2H), 1.74-1.01 (m, 27H), 0.97 (d, 3H, $J = 6.4$), 0.93-0.81 (m, 12H).

$^{13}$C$^1$H NMR (101 MHz, CDCl$_3$) $\delta$ 148.0, 129.2, 115.2, 111.8, 48.6, 44.8, 39.4, 37.5-37.3, 34.3, 34.2, 32.8, 31.2, 28.0, 24.8, 24.5, 24.4, 22.7, 22.6, 19.8-19.7, 12.4.
4-[Ethyl-phenyl-amino]butan-1-ol (Table 2, Entry 7)

\[
\text{\begin{array}{c}
\text{N} \\
\text{-} \\
\text{OH}
\end{array}}
\]

The compound was prepared as described using the procedure A (m = 43 mg, 89% isolated yield).

\(^{1}\text{H NMR (400 MHz, CDCl}_3\) \(\delta 7.26-7.18\) (m, 2H), 6.75-6.65 (m, 3H), 3.67 (t, 2H, \(J = 6.3\)), 3.36 (q, 2H, \(J = 7.2\)), 3.29 (t, 2H, \(J = 7.2\)), 1.96 (br s, 1H), 1.64-1.57 (m, 4H), 1.16 (t, 3H, \(J = 7.2\)).

\(^{13}\text{C\{(1\text{H)} NMR (101 MHz, CDCl}_3\) \(\delta 148.0, 129.3, 116.1, 112.7, 62.8, 50.4, 45.4, 30.5, 24.2, 12.2,\)}

MS (EI): m/z: 239 (12, M\(^+\)), 134 (100), 120 (12), 106 (27), 91 (16), 77 (18).

N-Hexyl-2,3-dihydro-1H-indole (Table 2, Entry 12)

\[
\text{\begin{array}{c}
\text{N} \\
\text{-} \\
\text{OH}
\end{array}}
\]

The compound was prepared as described using the procedure A (m = 41 mg, 81% isolated yield).

\(^{1}\text{H NMR (400 MHz, CDCl}_3\) \(\delta 7.10-7.03\) (m, 2H), 6.68-6.60 (m, 1H), 6.46 (d, 1H, \(J = 7.4\)), 3.34 (t, 2H, \(J = 8.2\)), 3.04 (t, 2H, \(J = 7.2\)), 2.96 (t, 2H, \(J = 8.2\)), 1.72-1.51 (m, 2H), 1.49-1.22 (m, 6H), 0.92 (t, 3H, \(J = 6.2\)).

\(^{13}\text{C\{(1\text{H)} NMR (101 MHz, CDCl}_3\) \(\delta 152.7, 130.0, 127.3, 124.4, 117.3, 106.9, 53.1, 49.4, 31.8, 28.6, 27.3, 27.0, 22.7, 14.1.\)}

N-(3-Phenylpropyl)indoline (Table 2, Entry 13)

\[
\text{\begin{array}{c}
\text{N} \\
\text{-} \\
\text{OH}
\end{array}}
\]

The compound was prepared as described using the procedure A (m = 41 mg, 70% isolated yield).

\(^{1}\text{H NMR (400 MHz, CDCl}_3\) \(\delta 7.35-7.27\) (m, 2H), 7.24-7.17 (m, 3H), 7.02 (t, 1H, \(J = 7.6\)), 6.55 (t, 1H, \(J = 7.6\)), 6.42 (d, 1H, \(J = 8.0\)), 3.36 (t, 2H, \(J = 8.2\)), 3.09 (t, 2H, \(J = 7.0\)), 2.98 (t, 2H, \(J = 8.2\)), 2.75 (t, 2H, \(J = 7.8\)), 1.95 (quint, 2H, \(J = 7.4\)).

\(^{13}\text{C\{(1\text{H)} NMR (75 MHz, CDCl}_3\) \(\delta 151.7, 140.9, 129.0, 127.5, 127.4, 126.3, 124.8, 123.4, 116.4, 105.9, 52.1, 47.7, 32.3, 28.1, 27.6.\)}

N-(3-Phenylpropyl)-1,2,3,4-tetrahydroquinoline (Table 2, Entry 14)

\[
\text{\begin{array}{c}
\text{N} \\
\text{-} \\
\text{OH}
\end{array}}
\]

The compound was prepared as described using the procedure A (m = 60 mg, 96% isolated yield).

\(^{1}\text{H NMR (400 MHz, CDCl}_3\) \(\delta 7.34-7.27\) (m, 2H), 7.24-7.17 (m, 3H), 7.02 (t, 1H, \(J = 7.6\)), 6.94 (d, 1H, \(J = 7.2\)), 6.55 (t, 1H, \(J = 7.2\)), 6.50 (d, 1H, \(J = 8.0\)), 3.32-3.23 (m, 4H), 2.76 (t, 2H, \(J = 6.2\)), 2.67 (t, 2H, \(J = 7.8\)), 2.00-1.89 (m, 4H).

\(^{13}\text{C\{(1\text{H)} NMR (101 MHz, CDCl}_3\) \(\delta 145.3, 141.8, 129.1, 128.34, 128.32, 127.0, 125.8, 122.3, 115.4, 110.5, 50.9, 49.4, 33.4, 28.2, 27.7, 22.3.\)}
N-Benzyl-N-(3-phenylpropyl)aniline (Table 2, Entry 15)

The compound was prepared as described using the procedure A (m = 59 mg, 75% isolated yield).  
$^1$H NMR (400 MHz, CDCl$_3$) δ 7.35-7.27 (m, 3H), 7.25-7.14 (m, 9H), 6.71-6.61 (m, 3H), 4.55 (s, 2H), 3.44 (t, 2H, $J = 7.6$), 2.67 (t, 2H, $J = 7.6$), 2.08-1.95 (m, 2H).  
$^{13}$C{$_1$H} NMR (75 MHz, CDCl$_3$) δ 148.6, 141.7, 139.1, 129.2, 128.6, 128.44, 128.37, 126.8, 126.6, 126.0, 116.2, 112.3, 54.6, 50.8, 33.4, 28.6.  
MS (EI): m/z: 301 (10, M$^+$), 196 (23), 106 (6), 91 (100), 77 (9).

N-Hexylaniline (Table 4, Entry 1)$^1$

The compound was prepared as described using the procedure B (m = 29 mg, 88% isolated yield).  
$^1$H NMR (300 MHz, CDCl$_3$) δ 7.18 (t, 2H, $J = 7.3$), 6.69 (t, 1H, $J = 7.3$), 6.61 (d, 2H, $J = 7.3$), 3.63 (br s, 1H), 3.11 (t, 2H, $J = 7.2$), 1.62 (m, 2H), 1.52-1.21 (m, 6H), 0.91 (t, 3H, $J = 6.4$).  
$^{13}$C{$_1$H} NMR (75 MHz, CDCl$_3$) δ 148.6, 129.2, 117.1, 112.7, 44.0, 31.7, 29.6, 26.9, 22.6, 14.1.

N-Hexyl-4-methoxyaniline (Table 4, Entry 2)$^6$

The compound was prepared as described using the procedure C (m = 37 mg, 72% isolated yield).  
$^1$H NMR (300 MHz, CDCl$_3$) δ 6.78 (d, 2H, $J = 9.0$), 6.58 (d, 2H, $J = 9.0$), 3.80 (br s, 1H), 3.75 (s, 3H), 3.06 (t, 2H, $J = 7.2$), 1.60 (quint, 2H, $J = 7.1$), 1.48-1.14 (m, 6H), 0.90 (t, 3H, $J = 6.8$).  
$^{13}$C{$_1$H} NMR (75 MHz, CDCl$_3$) δ 152.0, 142.9, 114.9, 114.0, 55.9, 45.1, 31.7, 29.7, 26.9, 22.7, 14.1.

N-(3-Phenylpropyl)aniline (Table 4, Entry 3)$^1$

The compound was prepared as described using the procedure B (m = 41 mg, 81% isolated yield).  
$^1$H NMR (400 MHz, CDCl$_3$) δ 7.36-7.14 (m, 7H), 6.71 (t, 1H, $J = 7.2$), 6.59 (d, 2H, $J = 8.0$), 3.63 (br s, 1H), 3.17 (t, 2H, $J = 7.0$), 2.76 (t, 2H, $J = 7.6$), 2.0-1.9 (m, 2H).  
$^{13}$C{$_1$H} NMR (101 MHz, CDCl$_3$) δ 148.4, 141.7, 129.3, 128.5, 128.4, 126.0, 117.3, 112.8, 43.5, 33.5, 31.1.
N-(3-Phenylpropyl)-4-fluoro-aniline (Table 4, Entry 4)

The compound was prepared as described using the procedure B (m = 39 mg, 68% isolated yield).

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.31 (m, 2H), 7.25-7.17 (m, 3H), 6.93-6.84 (m, 2H), 6.56-6.46 (m, 2H), 3.48 (br s, 1H), 3.12 (t, 2H, $J$ = 7.0), 2.75 (t, 2H, $J$ = 7.4), 2.0-1.9 (m, 2H).

$^{13}$C{$^1$H} NMR (101 MHz, CDCl$_3$) $\delta$ 155.8 (d, $J_{CF}$ = 234.6) 144.7, 141.6, 128.5 (d, $J_{CF}$ = 6.8), 126.0, 115.6 (d, $J_{CF}$ = 22.3), 113.6 (d, $J_{CF}$ = 7.3), 44.2, 33.4, 31.1.

MS (EI): m/z: 229 (18, M$^+$), 124 (100), 111 (13), 91 (13), 77 (6).

N-(3-Phenylpropyl)-4-bromo-aniline (Table 4, Entry 5)

The compound was prepared as described using the procedure B (m = 55 mg, 76% isolated yield).

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.34-7.16 (m, 7H), 6.43 (d, 2H, $J$ = 8.4), 3.62 (br s, 1H), 3.11 (t, 2H, $J$ = 6.8), 2.73 (t, 2H, 7.5), 2.0-1.9 (m, 2H).

$^{13}$C{$^1$H} NMR (101 MHz, CDCl$_3$) $\delta$ 147.3, 141.5, 131.9, 128.5, 128.4, 126.1, 114.3, 108.7, 43.4, 33.4, 30.9.

MS (EI): m/z: 291 (35, M$^+$), 289 (37, M$^+$), 186 (91), 184 (100), 173 (16), 171 (17), 157 (5), 155 (5), 117 (16), 105 (51), 91 (67), 77 (24), 65 (27), 51 (15).

N-(3-Phenylpropyl)-4-methyl-aniline (Table 4, Entry 7)$^8$

The compound was prepared as described using the procedure C (m = 51 mg, 91% isolated yield).

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.32-7.25 (m, 2H), 7.22-7.15 (m, 3H), 6.99 (d, 2H, $J$ = 7.2), 6.55 (d, 2H, $J$ = 7.6), 3.48 (br s, 1H), 3.14 (t, 2H, $J$ = 7.0), 2.74 (t, 2H, $J$ = 7.6), 2.25 (s, 3H), 2.01-1.89 (m, 2H).

$^{13}$C{$^1$H} NMR (75 MHz, CDCl$_3$) $\delta$ 146.1, 141.8, 129.8, 128.5, 126.5, 126.0, 113.1, 43.9, 33.5, 31.2, 20.5.

N-(3-Phenylpropyl)-4-methoxy-aniline (Table 4, Entry 8)$^7$

The compound was prepared as described using the procedure C (m = 40 mg, 66% isolated yield).

$^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 7.38-7.13 (m, 5H), 6.79 (d, 2H, $J$ = 8.8), 6.56 (d, 2H, $J$ = 8.7), 3.76 (s, 3H), 3.12 (t, 2H, $J$ = 6.9), 2.75 (t, 2H, $J$ = 7.7), 2.0-1.9 (m, 2H).

$^{13}$C{$^1$H} NMR (75 MHz, CDCl$_3$) $\delta$ 152.1, 142.7, 141.8, 128.44, 128.43, 125.9, 115.0, 114.1, 55.9, 44.5, 33.5, 31.2.
N-(3-Phenylpropyl)naphthalen-1-amine (Table 4, Entry 9)

The compound was prepared as described using the procedure B (m = 48 mg, 74% isolated yield).

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.69 (d, 1H, $J = 7.6$), 7.59 (d, 1H, $J = 8.0$), 7.39-7.28 (m, 2H), 7.25-7.08 (m, 7H), 6.47 (d, 1H, $J = 7.6$), 4.20 (br s, 1H), 3.23 (t, 2H, $J = 6.8$), 2.74 (t, 2H, $J = 7.4$), 2.03 (quint, 2H, $J = 7.2$). $^{13}$C{$_1$H} NMR (101 MHz, CDCl$_3$) δ 143.4, 141.6, 134.3, 128.6, 128.5, 128.4, 126.6, 126.0, 125.6, 124.6, 123.3, 119.7, 117.2, 104.2, 43.7, 33.7, 30.8.

ESI-HR-MS: [M+H]$^+$ (C$_{19}$H$_{20}$N) Theoretical m/z: 262.1596 Found m/z: 262.1593.

N-(3-Phenylpropyl)-2,4,6-trimethyl-aniline (Table 4, Entry 10)

The compound was prepared as described using the procedure B (m = 45 mg, 71% isolated yield).

$^1$H NMR (400 MHz, CDCl$_3$) δ 7.34-7.28 (m, 2H), 7.24-7.17 (m, 3H), 6.83 (s, 2H), 2.99 (t, 2H, $J = 7.2$), 2.81 (br s, 1H), 2.74 (t, 2H, $J = 7.8$), 2.25 (s, 9H), 1.93 (m, 2H).

$^{13}$C{$_1$H} NMR (101 MHz, CDCl$_3$) δ 143.6, 141.9, 131.2, 129.6, 129.4, 128.4, 125.9, 48.5, 33.6, 32.9, 20.7, 18.4.

ESI-HR-MS: [M+H]$^+$ (C$_{18}$H$_{24}$N) Theoretical m/z: 254.1909 Found m/z: 254.1907.

N-(3-Phenylpropyl)-2,6-diisopropylaniline (Table 4, Entry 11)

The compound was prepared as described using the procedure B (m = 56 mg, 76% isolated yield).

$^1$H NMR (300 MHz, CDCl$_3$) δ 7.35-7.27 (m, 2H), 7.25-7.16 (m, 3H), 7.13-6.98 (m, 3H), 3.32-3.13 (m, 2H), 2.90 (t, 2H, $J = 7.1$), 2.78 (t, 2H, $J = 7.7$), 2.10-1.88 (m, 2H), 1.22 (d, 12H, $J = 7.0$).

$^{13}$C{$_1$H} NMR (75 MHz, CDCl$_3$) δ 145.4, 129.7, 125.9, 123.6, 123.5, 51.5, 33.5, 32.5, 27.7, 24.2.

ESI-HR-MS: [M+H]$^+$ (C$_{21}$H$_{30}$N) Theoretical m/z: 296.2378 Found m/z: 296.2379.

N-(sec-Butyl)-4-methylaniline (Table 4, Entry 12)

The compound was prepared as described using the procedure D (m = 38 mg, 91% isolated yield).

$^1$H NMR (300 MHz, CDCl$_3$) δ 7.02 (d, 2H, $J = 8.4$), 6.55 (d, 2H, $J = 8.4$), 3.49-3.33 (m, 1H), 3.32 (br s, 1H), 2.28 (s, 3H), 1.75-1.40 (m, 2H), 1.20 (d, 3H, $J = 6.3$), 0.99 (t, 3H, $J = 7.4$).

$^{13}$C{$_1$H} NMR (75 MHz, CDCl$_3$) δ 145.4, 129.7, 125.9, 113.3, 50.1, 29.6, 20.3, 20.2, 10.3.
4-(Phenylamino)butan-1-ol (Table 4, Entry 13)\(^\text{10}\)

![Chemical structure](image)

The compound was prepared as described using the procedure B (m = 29 mg, 71% isolated yield).  
\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.18 (t, 2H, \(J = 7.4\)), 6.70 (t, 1H, \(J = 7.2\)), 6.63 (d, 2H, \(J = 8.0\)), 3.70 (t, 2H, \(J = 5.6\)), 3.16 (t, 2H, \(J = 5.6\)), 2.63 (br s, 2H), 1.71 (m, 4H).

\(^{13}\)C\({}^1\)H NMR (100 MHz, CDCl\(_3\)) \(\delta\) 148.3, 129.2, 117.5, 112.9, 62.7, 43.9, 30.4, 26.1.

4-(p-Tolylamino)butan-1-ol (Table 4, Entry 14)\(^\text{11}\)

![Chemical structure](image)

The compound was prepared as described using the procedure B (m = 32 mg, 72% isolated yield).  
\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 6.99 (d, 2H, \(J = 7.6\)), 6.55 (d, 2H, \(J = 7.6\)), 3.69 (m, 2H), 3.14 (m, 2H), 2.58 (br s, 2H), 2.23 (s, 3H), 1.70 (m, 4H).

\(^{13}\)C\({}^1\)H NMR (75 MHz, CDCl\(_3\)) \(\delta\) 146.1, 146.0, 129.8, 129.7, 126.8, 113.3, 62.7, 44.4, 30.5, 26.3, 20.4.

4-[(4-Bromophenyl)amino]butan-1-ol (Table 4, Entry 15)

![Chemical structure](image)

The compound was prepared as described using the procedure B (m = 56 mg, 92% isolated yield).  
\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.24 (d, 2H, \(J = 8.8\)), 6.47 (d, 2H, \(J = 8.8\)), 3.68 (t, 2H, \(J = 5.5\)), 3.11 (t, 2H, \(J = 7.0\)), 2.62 (br s, 2H), 1.68 (m, 4H).

\(^{13}\)C\({}^1\)H NMR (101 MHz, CDCl\(_3\)) \(\delta\) 147.3, 131.9, 114.4, 108.8, 62.6, 43.9, 30.2, 25.9.

MS (EI): m/z: 245 (25, M+), 243 (25, M+), 186 (93), 129 (53), 115 (18), 77 (10).

4-(Naphthalen-1-ylamino)butan-1-ol (Table 4, Entry 16)

![Chemical structure](image)

The compound was prepared as described using the procedure B (m = 33 mg, 61% isolated yield).  
\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.87-7.75 (m, 2H), 7.49-7.39 (m, 2H), 7.35 (t, 1H, \(J = 7.5\)), 7.26-7.20 (m, 1H), 6.62 (d, 1H, \(J = 7.6\)), 3.75 (t, 2H, \(J = 6.0\)), 3.33 (t, 2H, \(J = 6.8\)), 1.95-1.83 (m, 2H), 1.83-1.72 (m, 2H).

\(^{13}\)C\({}^1\)H NMR (101 MHz, CDCl\(_3\)) \(\delta\) 143.5, 134.3, 128.7, 126.6, 125.7, 124.7, 123.5, 119.8, 117.4, 104.4, 62.7, 44.1, 30.6, 25.9.

MS (EI): m/z: 215 (36, M+), 156 (100), 143 (11), 129 (53), 115 (18), 77 (10).
4-(Mesitylamino)butan-1-ol (Table 4, Entry 17)

\[
\begin{align*}
\text{H} & \quad \text{N} & \quad \text{H} & \quad \text{OH} \\
\end{align*}
\]

The compound was prepared as described using the procedure B (m = 33 mg, 64% isolated yield). 

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta \) 6.82 (s, 2H), 3.68 (t, 2H, \(J = 6.2\)), 2.94 (t, 2H, \(J = 6.2\)), 2.59 (br s, 2H), 2.26 (s, 6H), 2.23 (s, 3H), 1.71 (m, 4H).

\(^{13}\)C\({\text{\textsuperscript{1}}\text{H}}\) NMR (75 MHz, CDCl\(_3\)) \(\delta \) 143.1, 131.8, 129.9, 129.5, 62.9, 48.9, 31.0, 28.3, 20.6, 18.3.

MS (EI): \(m/z\) 207 (13, M\(^+\)), 148 (100), 134 (8), 119 (10), 91 (10), 77 (6).

References

$^1$H NMR and $^{13}$C NMR spectra

N-Ethyl-N-hexylaniline (Table 3, Entries 1, 2, 10)
N-Butyl-N-ethylaniline (Table 3, Entries 3, 9)
N-Ethyl-N-isopentylaniline (Table 3, Entry 4, 11)
N-Ethyl-N-isobutylaniline (Table 3, Entry 5)

 ppm (t1)

 Frequency (MHz):
 (f1) 300.132
 Original Points Count:
 (f1) 16384
 Actual Points Count:
 (f1) 32768
 Acquisition Time (sec): 
 (f1) 2.6542
 Spectral Width (ppm): 
 (f1) 20.567
 Pulse Program:
 ZG30
 Temperature:
 296.8733
 Number of Scans:
 16

 ppm (t1)

 Frequency (MHz):
 (f1) 75.475
 Original Points Count:
 (f1) 32768
 Actual Points Count:
 (f1) 65536
 Acquisition Time (sec):
 (f1) 1.8219
 Spectral Width (ppm):
 (f1) 238.298
 Pulse Program:
 ZGPG30
 Temperature:
 298.1003
 Number of Scans:
 500

 ppm (t1)

 Frequency (MHz):
 (f1) 76.475
 Original Points Count:
 (f1) 32768
 Actual Points Count:
 (f1) 65536
 Acquisition Time (sec):
 (f1) 1.8219
 Spectral Width (ppm):
 (f1) 238.298
 Pulse Program:
 ZGPG30
 Temperature:
 298.1003
 Number of Scans:
 500

 ppm (t1)

 Frequency (MHz):
 (f1) 300.132
 Original Points Count:
 (f1) 16384
 Actual Points Count:
 (f1) 32768
 Acquisition Time (sec):
 (f1) 2.6542
 Spectral Width (ppm):
 (f1) 20.567
 Pulse Program:
 ZG30
 Temperature:
 296.8733
 Number of Scans:
 16

 ppm (t1)

 Frequency (MHz):
 (f1) 75.475
 Original Points Count:
 (f1) 32768
 Actual Points Count:
 (f1) 65536
 Acquisition Time (sec):
 (f1) 1.8219
 Spectral Width (ppm):
 (f1) 238.298
 Pulse Program:
 ZGPG30
 Temperature:
 298.1003
 Number of Scans:
 500

 ppm (t1)
N-Ethyl-N-(3-phenylpropyl)aniline (Table 3, Entry 6)
4-[Ethyl(phenyl)amino]butan-1-ol (Table 3, Entry 7)

![Chemical Structure]

**Frequency (MHz):**
- $f_1$: 400.162
- $f_2$: 100.631

**Original Points Count:**
- $f_1$: 32768
- $f_2$: 32768

**Actual Points Count:**
- $f_1$: 65536
- $f_2$: 32768

**Acquisition Time (sec):**
- $f_1$: 3.9846
- $f_2$: 1.2583

**Spectral Width (ppm):**
- $f_1$: 20.551
- $f_2$: 258.783

**Pulse Program:**
- ZG30
- ZGPG30

**Temperature:**
- 299.1094
- 299.1232

**Number of Scans:**
- 8
- 1024
N-Ethyl-N-(3,7,11,15-tetramethyhexadecyl)aniline (Table 3, Entry 8)

Spectrum Title: cdlhq684
Frequency (MHz): (f 1) 300.132
Original Points Count: (f 1) 16384
Actual Points Count: (f 1) 32768
Acquisition Time (sec): (f 1) 2.6542
Spectral Width (ppm): (f 1) 20.567
Pulse Program: ZG30
Temperature: 295.4009
Number of Scans: 8

Spectrum Title: cdlhq684
Frequency (MHz): (f 1) 100.613
Original Points Count: (f 1) 32768
Actual Points Count: (f 1) 32768
Acquisition Time (sec): (f 1) 1.3631
Spectral Width (ppm): (f 1) 238.921
Pulse Program: Unknown
Temperature: 295.4009
Number of Scans: 8
N-Hexylindoline (Table 3, Entry 12)
N-(3-Phenylpropyl)indoline (Table 3, Entry 13)
N-(3-Phenylpropyl)-1,2,3,4-tetrahydroquinoline (Table 3, Entry 14)
**N-Benzyl-N-(3-phenylpropyl)aniline (Table 3, Entry 15)**

![N-Benzyl-N-(3-phenylpropyl)aniline](image)

**Spectroscopic Data**

- **Frequency (MHz):**
  - (f 1) 100.623
  - (f 1) 400.162

- **Original Points Count:**
  - (f 1) 32768
  - (f 1) 32768

- **Actual Points Count:**
  - (f 1) 65536
  - (f 1) 65536

- **Acquisition Time (sec):**
  - (f 1) 1.3631
  - (f 1) 3.9846

- **Spectral Width (ppm):**
  - (f 1) 238.897
  - (f 1) 20.551

- **Pulse Program:**
  - ZGPG30
  - ZG30

- **Temperature:**
  - 299.3946
  - 299.1041

- **Number of Scans:**
  - 512
  - 16

*H2O
**N-Hexylaniline (Table 4, Entry 1)**

\[
\text{\textbf{N-Hexylaniline}}
\]

**Frequency (MHz):**
(f 1) 75.475

**Original Points Count:**
(f 1) 32768

**Acquisition Time (sec):**
(f 1) 1.8219

**Spectral Width (ppm):**
(f 1) 238.298

**Pulse Program:**
ZGPG30

**Temperature:**
296.996

**Number of Scans:**
102

---

**Frequency (MHz):**
(f 1) 300.132

**Original Points Count:**
(f 1) 16384

**Actual Points Count:**
(f 1) 32768

**Acquisition Time (sec):**
(f 1) 2.6542

**Spectral Width (ppm):**
(f 1) 20.567

**Pulse Program:**
ZG30

**Temperature:**
296.6279

**Number of Scans:**
16

---

**Frequency (MHz):**
(f 1) 400.120

**Original Points Count:**
(f 1) 2048

**Actual Points Count:**
(f 1) 4096

**Acquisition Time (sec):**
(f 1) 4.307

**Spectral Width (ppm):**
(f 1) 4.95

**Pulse Program:**
ZG30

**Temperature:**
296.6279

**Number of Scans:**
6
N-Hexyl-4-methoxyaniline (Table 4, Entry 2)

\[
\text{MeO}^+ \\
\text{H} \\
\text{N} \\
\text{C}_6\text{H}_{13} \\
\]

Frequency (MHz):
(f 1) 75.475
Original Points Count:
(f 1) 32768
Actual Points Count:
(f 1) 65536
Acquisition Time (sec):
(f 1) 1.8219
Spectral Width (ppm):
(f 1) 238.298
Pulse Program:
ZGPG30
Temperature:
297.3641
Number of Scans:
500

Frequency (MHz):
(f 1) 300.132
Original Points Count:
(f 1) 16384
Actual Points Count:
(f 1) 32768
Acquisition Time (sec):
(f 1) 2.6542
Spectral Width (ppm):
(f 1) 20.567
Pulse Program:
ZG30
Temperature:
295.8917
Number of Scans:
16
N-(3-Phenylpropyl)aniline (Table 4, Entry 3)

\[
\begin{align*}
\text{Frequency (MHz):} & \quad f_1 = 75.475 \\
\text{Original Points Count:} & \quad f_1 = 32768 \\
\text{Actual Points Count:} & \quad f_1 = 65536 \\
\text{Acquisition Time (sec):} & \quad f_1 = 1.8219 \\
\text{Spectral Width (ppm):} & \quad f_1 = 238.298 \\
\text{Pulse Program:} & \quad ZGPG30 \\
\text{Temperature:} & \quad 296.7506 \\
\text{Number of Scans:} & \quad 500
\end{align*}
\]
4-Fluoro-N-(3-phenylpropyl)aniline (Table 4, Entry 4)
4-Bromo-N-(3-phenylpropyl)aniline (Table 4, Entry 5)
4-Methyl-N-(3-phenylpropyl)aniline (Table 4, Entry 7)

\[ \text{Structure Image} \]

**Spectral Data**
- **Frequency (MHz):**
  - (f1) 400.162
  - (f1) 100.630
- **Original Points Count:**
  - (f1) 32768
  - (f1) 32768
- **Actual Points Count:**
  - (f1) 65536
  - (f1) 65536
- **Acquisition Time (sec):**
  - (f1) 3.9846
  - (f1) 1.3631
- **Spectral Width (ppm):**
  - (f1) 20.551
  - (f1) 238.879
- **Pulse Program:**
  - ZG30
  - ZGPG30
- **Temperature:**
  - 299.0839
  - 299.1105
- **Number of Scans:**
  - 8
  - 256

**Graphs**: Two NMR spectra are shown, one for each frequency setting, displaying the chemical shifts and intensity at various ppm values.
4-Methoxy-N-(3-phenylpropyl)aniline (Table 4, Entry 8)
N-(3-Phenylpropyl)naphthalen-1-amine (Table 4, Entry 9)
N-(3-phenylpropyl)-2,4,6-trimethyl aniline (Table 4, Entry 10)

\[
\begin{align*}
\text{N-} & \text{(3-phenylpropyl)-2,4,6-trimethyl aniline (Table 4, Entry 10)} \\
\end{align*}
\]
2,6-Diisopropyl-N-(3-phenylpropyl)aniline (Table 4, Entry 11)

Chemical structure image

Frequency (MHz): (f1) 300.132
Original Points Count: (f1) 16384
Actual Points Count: (f1) 32768
Acquisition Time (sec): (f1) 2.6542
Spectral Width (ppm): (f1) 20.567
Pulse Program: ZG30
Temperature: 296.5052
Number of Scans: 8

Frequency (MHz): (f1) 100.613
Original Points Count: (f1) 32768
Actual Points Count: (f1) 32768
Acquisition Time (sec): (f1) 1.3631
Spectral Width (ppm): (f1) 238.921
Pulse Program: Unknown
N-(sec-Butyl)-4-methylaniline (Table 4, Entry 12)
4-(phenylamino)butan-1-ol (Table 4, Entry 13)

\[
\begin{align*}
\text{Original Points Count:} & \quad 65536 \\
\text{Actual Points Count:} & \quad 32768 \\
\text{Acquisition Time (sec):} & \quad 1.8219 \\
\text{Spectral Width (ppm):} & \quad 238.298 \\
\text{Pulse Program:} & \quad ZGPG30 \\
\text{Temperature:} & \quad 295.8917 \\
\text{Number of Scans:} & \quad 41
\end{align*}
\]

\[
\begin{align*}
\text{Frequency (MHz):} & \quad 75.475 \\
\text{Original Points Count:} & \quad 65536 \\
\text{Actual Points Count:} & \quad 32768 \\
\text{Acquisition Time (sec):} & \quad 3.9846 \\
\text{Spectral Width (ppm):} & \quad 20.551 \\
\text{Pulse Program:} & \quad ZG30 \\
\text{Temperature:} & \quad 299.1143 \\
\text{Number of Scans:} & \quad 8
\end{align*}
\]
4-(p-Tolylamino)butan-1-ol (Table 4, Entry 14)
4-[(4-Bromophenyl)amino]butan-1-ol (Table 4, Entry 15)
4-(Naphthalen-1-ylamino)butan-1-ol (Table 4, Entry 16)
4-(Mesitylamino)butan-1-ol (Table 4, Entry 17)

\[
\text{[Chemical Structure Image]}
\]

**Frequency (MHz):**
- (f 1) 400.160
- (f 2) 75.475

**Original Points Count:**
- (f 1) 65536
- (f 2) 32768

**Actual Points Count:**
- (f 1) 65536
- (f 2) 65536

**Acquisition Time (sec):**
- (f 1) 7.9692
- (f 2) 1.8219

**Spectral Width (ppm):**
- (f 1) 20.551
- (f 2) 238.298

**Pulse Program:**
- Unknown
- ZGPG30

**Temperature:**
- 295.8917

**Number of Scans:**
- 1500

**ppm (t1):**
- 0.0
- 1.0
- 2.0
- 3.0
- 4.0
- 5.0
- 6.0
- 7.0
- 8.0

**ppm (t2):**
- 0
- 50
- 100
- 150
- 200
- 250

**Chemical Shifts:**

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<th>ppm (t1)</th>
<th>ppm (t2)</th>
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<tr>
<td>1.709</td>
<td>1.709</td>
</tr>
</tbody>
</table>

**Note:**
- The table and spectrum images depict the chemical shifts and spectral data for 4-(Mesitylamino)butan-1-ol.
- The spectrum is shown with peaks at various ppm values, indicating the presence of different functional groups.
- The chemical structure is shown with the functional groups identified.

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