PART B

Reductive amination of aldehydes and ketones under microwave irradiation
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VB. 1. INTRODUCTION TO REDUCTIVE AMINATION OF ALDEHYDES AND KETONES

Reductive amination (also known as reductive alkylation) is a chemical reaction which involves the conversion of a carbonyl group to an amine, via an intermediate imine. The carbonyl group is most commonly a ketone or an aldehyde Scheme VB.1.

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

\[
\text{R} \quad \text{C} \quad \text{N} \quad \text{R} \quad \text{R} \\
\text{R} \quad \text{H} \\
\text{H} \\
\]

\[
\text{R} \quad \text{H} \\
\text{R} \\
\]

Scheme VB.1.

In this organic reaction, the amine first reacts with the carbonyl group to form a hemiaminal species, which subsequently loses one molecule of water in a reversible manner by alkylimino-de-oxo-bisubstitution, to form the imine. The equilibrium between aldehyde/ketone and imine can be shifted toward imine formation by removal of the formed water through physical or chemical means. This intermediate imine can then be isolated and reduced with a suitable reducing agent (e.g. sodium borohydride). This is indirect reductive amination. However, it is also possible to carry out the same reaction all in one pot, with the imine formation and reduction occurring concurrently. This is known as direct reductive amination, and is carried out with reducing agents that are more reactive toward protonated imines than ketones, and which are stable under moderately acidic conditions. These include sodium cyanoborohydride (NaBH₃CN, the Birch Reduction) and sodium triacetoxyborohydride (NaBH(OOCCH₃)₃).[1] This reaction has in recent years been performed in an aqueous environment casting doubt on the necessity of forming the imine. This is because the loss of the water molecule is thermodynamically disfavoured by the presence of a large amount of water in its environment, as seen in the work of Turner et al. This therefore, suggests that in some cases the reaction proceeds via direct reduction of the hemiaminal species.


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This reaction is related to the Eschweiler-Clarke reaction in which amines are methylated to tertiary amines, the Leuckart-Wallach reaction with formic acid and to other amine alkylation methods as the Mannich reaction and the Petasis reaction.

The direct reductive amination (DRA) of aldehydes and ketones is one of the most widely used routes to secondary and tertiary amines. Secondary and tertiary amines find application as intermediates in organic synthesis, biologically active and pharmaceutically relevant compounds.[2] In reductive amination, aromatic amines and aliphatic carbonyls together give highly reactive and unstable imine intermediates; this reduces the need to isolate imines, which is the major advantage of this reaction.[3]


Reactive amination under microwave irradiation
VB. 2. SOME INTERESTING AND USEFUL EXAMPLES FROM LITERATURE

Sodium triacetoxyborohydride is a general, mild, and selective reducing agent for the reductive amination of various aldehydes and ketones. 1,2-Dichloroethane (DCE) is the preferred reaction solvent, but reactions can also be carried out in tetrahydrofuran and occasionally in acetonitrile. Acetic acid may be used as catalyst with ketone reactions. Acid sensitive functional groups such as acetals and ketals, and reducible functional groups such as C-C multiple bonds and cyano and nitro groups are tolerated (Scheme VB.2).[4]

\[
\begin{align*}
\text{R}^1\text{C}=\text{O} + \text{HN}^3 & \xrightarrow{1.3-1.6\text{ eq. NaBH(OAc)}_3} \text{RN}^3 \\
& \xrightarrow{1.2\text{ eq. AcOH}} \text{DCE or THF, RT, 0.5-75 h}
\end{align*}
\]

Scheme VB.2.

In the reductive amination of some aldehydes with primary amines where dialkylation is a problem, a stepwise procedure involving imine formation in MeOH followed by reduction with NaBH₄ was developed (Scheme VB.3).[4]

\[
\begin{align*}
\text{R}^1\text{C}=\text{O} + \text{H}_2\text{N}^1 & \xrightarrow{\text{MeOH, RT, } \sim 3\text{ h}} \text{R}^1\text{N}^1 \\
& \xrightarrow{1.6\text{ eq. NaBH}_4} \text{10-15 min}
\end{align*}
\]

Scheme VB.3.

Aldehydes and ketones were easily converted to the corresponding amines by the reaction of amines in methanol using decaborane (B₁₀H₁₄) at room temperature under nitrogen. The reaction is simple and efficient (Scheme VB.4).[5]

Scheme VB.4.

A simple and convenient procedure allows the reductive amination of aldehydes and ketones using sodium borohydride as reducing agent and boric acid, p-toluenesulfonic acid monohydrate or benzoic acid as activator under solvent-free conditions (Scheme VB.5).[6]

Scheme VB.5.

An effective reductive alkylation of electron-deficient o-chloroarylamines was developed. The derived N-alkylated o-chloroarylamines were elaborated to N-alkylazaindololes and N-alkylindoles via a novel one-pot process comprising copper-free Sonogashira alkynylation and a base-mediated indolization reaction (Scheme VB.6).[7]

Scheme VB.6.

An efficient methodology for the reductive alkylation of secondary amines with aldehydes and Et$_3$SiH using an iridium complex as a catalyst has been developed. In addition, a cheaper, easy-to-handle, and environmentally friendly reducing reagent such as polymethylhydrosiloxane (PMHS) in place of Et$_3$SiH was also useful (Scheme VB.7).[8]

$$\text{R-} \overset{\text{NH}}{\text{N}} \text{H} + \overset{\text{O}}{\text{H}} \overset{\text{O}}{\text{H}} \text{H} \rightarrow \overset{\text{R}}{\text{R}} \overset{\text{N}}{\text{N}} \text{R}$$

Scheme VB.7.

α-Imino esters derived from aryl and alkyl keto esters could be reduced to the corresponding α-amino esters in excellent yields and in high enantiomeric excesses using 5 mol-% of a chiral phosphoric acid as catalyst, Hantzsch ester as hydride donor, and toluene as solvent (Scheme VB.8).[9]

$$\text{COOEt} \overset{\text{R}}{\text{R}} \text{COOEt} + \overset{\text{H}_2\text{N}}{\text{PMP}} \rightarrow \overset{\text{R}}{\text{PMP}} \text{COOEt}$$

Scheme VB.8.


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A biomimetic direct reductive amination of ketones relies on selective imine activation by hydrogen bond formation with thiourea as hydrogen bond donor and utilizes the Hantzsch ester for transfer hydrogenation. The method allows the efficient synthesis of structurally diverse amines (Scheme VB.9).[10]

![Scheme VB.9.](image)

Treatment of ketones with ammonia in ethanol and titanium(IV) isopropoxide, followed by in situ reduction with sodium borohydride allows a highly chemoselective reductive mono-alkylation of ammonia. A simple workup afforded primary amines in good to excellent yields. Reductive alkylation of ammonia with aldehydes afforded the corresponding symmetrical secondary amines selectively (Scheme VB.10).[11]

![Scheme VB.10.](image)

$N$-Alkylaminobenzenes were prepared in a simple and efficient one-pot synthesis by reduction of nitrobenzenes followed by reductive amination with decaborane ($\text{B}_{10}\text{H}_{14}$) in the presence of 10% Pd/C (Scheme VB.11).[12]

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$$\text{R, } R^1 = \text{alkyl, Ar}$$

Scheme VB.11.

**VB. 2.1. CONCLUSIONS**

In spite of improved methodology and benefits extended by the existing catalytic systems, there are still some limitations in substrate compatibility. Most of the reported methods for the reductive amination of aldehydes and ketones does not involve use of environmentally friendly reagents or catalysts, and hence, are not accepted in the concept of green chemistry. Hence developing a simple, convenient and economically viable protocol for the reductive amination of aldehydes and ketones using simple reducing agents is necessary.
VB. 3. PRESENT WORK: NaOEt: AN EFFECTIVE REAGENT FOR THE REDUCTIVE AMINATION OF ALDEHYDES AND KETONES UNDER MICROWAVE IRRADIATION

VB. 3.1. INTRODUCTION

In reductive amination, aromatic amines and aliphatic carbonyl compounds together give highly reactive and unstable imine intermediates; this reduces the need to isolate imines, which is the major advantage of the reductive amination reaction. To suppress undesirable reduction of starting carbonyl function, the choice of reductant becomes very critical, some of the recently developed reagents which effect direct reductive amination include: NaBH(OAc)₃,[13] ZnCl₂-NaBH₄,[14] NiCl₂-NaBH₄,[15] Ti(OiPr)₄-polymethylhydrosiloxane,[16] Ti(OiPr)₄-NaBH₄,[17] Bu₃SnH,[18] Bu₂SnClH and Bu₂SnIH,[19] decaborane,[20] silica gel-ZnBH₄,[21] Et₃SiH-trifluoroacetic acid,[22] pyridine-BH₃,[23] Et₃SiH-trifluoroacetic acid,[24] dibutyltin dichloride-phenylsilane in a nonprotic solvent,[25] triethyilsilane and an iridium compound is also reported.[26]


Reductive amination under microwave irradiation
Transiton metals and metal salts are also used for the direct reductive amination.[27] Most recently, Sajiki and co-workers described a selective monoalkylation of both aromatic and aliphatic amines using nitriles as an alkylating agents and Pd/C or Rh/C as catalysts.[28] In general, it is necessary to use excess of amines in order to prevent the competitive reduction of carbonyl group. All the reported methods involve use of expensive, complex and hazardous reagents and toxic organic solvents. On the other hand, microwave assisted chemistry has grabbed considerable amount of attention in recent years and has been successfully applied in various fields of synthetic organic chemistry.[29–33] it also finds application in the rapid preparation of radio-labeled materials,[31] in solvent-free reactions[32] and in phase-transfer catalysis.[33] It is very well understood that, for most chemical transformations requiring heat, microwave approaches can be used successfully. The main advantages of carrying out reactions under controlled microwave irradiation is that, the rate is enhanced significantly and good product yields are achieved.[34] Microwave assisted formation of imines and their reduction using either NaBH₄-CN, NaBH₄ or NaBH(OAc)₃ is known.[35– 37] But again these methods involve highly expensive reagents, and hence, we were interested in developing a simple, convenient and economically viable protocol for the reductive amination of aldehydes and ketones using simple reducing agents. In continuation of our work on synthesis of organic molecules under microwave irradiation,[38] 


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here we are presenting an efficient, simple and rapid reductive amination of aldehydes and Ketones to get secondary and tertiary amines using sodium ethoxide in a mixture of ethanol-THF under microwave irradiation. The process is mild and inexpensive, the yields are very high and the reactions go to completion with in 2–5 minutes as shown in (Scheme VB.12).

![Scheme VB.12.](image)

R = -H, -CH₃, -C₆H₅  
R² = -H, -CH₃  
R¹ = -OCH₃, 4-N(CH₃)₂  
R³ = -C₆H₅, 3-(Cl)C₆H₄, 3,4-(Cl)₂C₆H₃, -CH₂C₆H₅, C₆H₁₁

**VB. 3.2. RESULTS AND DISCUSSION**

In order to develop a new reagent system for the reductive amination of aldehydes and ketones, sodium ethoxide in ethanol-THF medium was selected. It was found that, this method did not require excess amount of amine compared to other methods and gave the intermediate imine instantaneously. Also, this reaction condition did not encourage the reduction of carbonyl compounds in the reaction mixture, whereas the conversion of the imine intermediates to the corresponding amines was easily accomplished. The absence of hydroxyl compound indicated that, the present reagent was able to discriminate between the reduction of the imine intermediate and the carbonyl compound present in the reaction mixture. Aromatic aldehydes with both electron-donating and electron-withdrawing substituents gave the corresponding products in the presence of respective amines easily under this condition, and the results of this study are presented in Table VB.1. It is clear from Table VB.1 that, a variety of aldehydes and ketones on treatment with primary and secondary amines in the presence of sodium ethoxide in ethanol-THF, afforded the corresponding amines in very high yields under microwave irradiation at 320 W in short duration. (Reactions were carried out at lower watts which took longer time to yield the product).
Table VB.1: Reductive amination of aldehydes and ketones with amines in presence of sodium ethoxide in ethanol-THF under microwave irradiation at 320 W.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Aldehyde(1)</th>
<th>Amine(2)</th>
<th>Product(3)$^a$</th>
<th>Time (min)</th>
<th>Yield (%)$^b$</th>
<th>M. P (°C) Observed</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>CHO</td>
<td>NH$_2$</td>
<td></td>
<td>3</td>
<td>88</td>
<td>35-38</td>
</tr>
<tr>
<td>b</td>
<td>CHO$_{Ome}$</td>
<td>NH$_2$</td>
<td></td>
<td>3</td>
<td>85</td>
<td>30-33</td>
</tr>
<tr>
<td>c</td>
<td>CHO$_{NMe_2}$</td>
<td>NH$_2$</td>
<td></td>
<td>4</td>
<td>80</td>
<td>oily liq.</td>
</tr>
<tr>
<td>d</td>
<td>CHO$_{Cl}$</td>
<td>NH$_2$</td>
<td></td>
<td>5</td>
<td>80</td>
<td>oily liq.</td>
</tr>
<tr>
<td>e</td>
<td>CHO$_{Cl}$</td>
<td>NH$_2$</td>
<td></td>
<td>4</td>
<td>79</td>
<td>oily liq.</td>
</tr>
<tr>
<td>f</td>
<td>CHO$_{Cl}$</td>
<td>NH$_2$</td>
<td></td>
<td>4</td>
<td>75</td>
<td>oily liq.</td>
</tr>
<tr>
<td>g</td>
<td>CHO</td>
<td>H$_2$N$_2$</td>
<td></td>
<td>4</td>
<td>90</td>
<td>oily liq.</td>
</tr>
<tr>
<td>h</td>
<td>CHO</td>
<td>NH$_2$</td>
<td></td>
<td>2</td>
<td>95</td>
<td>oily liq.</td>
</tr>
<tr>
<td>i</td>
<td>CHO$_{OCH_3}$</td>
<td>NH$_2$</td>
<td></td>
<td>5</td>
<td>80</td>
<td>oily liq.</td>
</tr>
<tr>
<td>j</td>
<td>CHO$_{O}$</td>
<td>NH$_2$</td>
<td></td>
<td>5</td>
<td>70</td>
<td>oily liq.</td>
</tr>
</tbody>
</table>

Reductive amination under microwave irradiation
| k | ![chemistry structure](image) | ![chemistry structure](image) | 2 | 97 | oily liq. |
| l | ![chemistry structure](image) | ![chemistry structure](image) | 2 | 97 | oily liq. |
| m | ![chemistry structure](image) | ![chemistry structure](image) | 2 | 98 | oily liq. |
| n | ![chemistry structure](image) | ![chemistry structure](image) | 2 | 98 | oily liq. |
| o | ![chemistry structure](image) | ![chemistry structure](image) | 3 | 96 | oily liq. |

*Characterized by IR and GC-mass spectral analysis and by comparison with authentic samples.

Isolated yields.

**VB. 3.3. EXPERIMENTAL**

All the reagents used were commercially available and all the solvents were distilled before use. THF was distilled and dried over sodium. All the reactions were carried out in a MILESTONE microwave reactor at 320 W. Reactions were monitored on TLC by comparison with the authentic samples. GC-Mass spectra were obtained using a Shimadzu GC-MS QP 5050A instrument equipped with a 30 m length and 0.32 mm dia BP-5 column with the column temperature 80–15–250 °C.

**VB. 3.4. GENERAL PROCEDURE FOR THE REDUCTIVE AMINATION OF ALDEHYDES AND KETONES UNDER MICROWAVE IRRADIATION**

A mixture of benzaldehyde (0.53 g, 5 mmol), piperidine (0.64 g, 7.5 mmol), sodium ethoxide [Prepared in situ from 0.115 g of atomized sodium metal in a mixture of EtOH-THF (1 mL-2 mL)] was taken in a Pyrex glass tube and placed in a microwave reactor. It was irradiated (320 W) for 2 minutes in intervals of 10 s. After completion of the reaction which was monitored by TLC (eluent: 8–10 % ethylacetate in light petrol). The organic matter was filtered, washed with saturated sodium bicarbonate solution (5 mL), water (5 mL) and then extracted with ether (5
mL × 3), dried over anhydrous sodium sulphate. Desired amine was obtained after the removal of solvent.

**VB. 3.5. MECHANISM**

A plausible mechanism for the direct reductive amination of carbonyl compounds with amines is envisaged. Ethoxide ion is expected to attack the carbonyl carbon to give intermediate \( A \), which on reaction with amine in the subsequent step, may give the iminium ion intermediate \( B \). \( B \) in presence of ethoxide ion is expected to get reduced to give the corresponding amine as shown in (Scheme VB.13).

![Scheme VB.13.](image)

**VB. 3.6. CONCLUSIONS**

In conclusion, a simple, convenient, cost effective approach for the one-pot reductive amination of aldehydes and ketones with amines using sodium ethoxide in ethanol-THF as solvent under microwave irradiation at 320 watts is developed. The reaction is facile, involves simple workup, uses readily available chemicals and gives high yield of the products in short reaction duration.
VB. 3.7. SPECTRAL DATA

**N-Benzylaniline** (3a)

IR (neat) v: 3419, 3026, 2924, 2853, 1949, 1602, 1505, 1324, 11267, 989, 749 cm\(^{-1}\). \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\): 4.02 (s, br, 1H, NH), 4.36 (s, 2H), 6.75–6.67 (m, 3H), 7.24–7.21 (t, \(J = 12\) Hz, 2H), 7.39–7.31 (m, 5H) ppm. \(^13\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\): 148.2, 139.5, 129.3, 128.7, 127.6, 127.3, 117.6, 113.0, 48.3 ppm.

**N-(4-Methoxybenzyl)aniline** (3b)

IR (neat) v: 3416, 3019, 2930, 2835, 1922, 1603, 1508, 1321, 1247, 1177, 1034, 824, 750, 692 cm\(^{-1}\). \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\): 3.74 (s, 3H, OCH\(_3\)), 3.88 (s, br, 1H, NH), 4.19 (s, 2H, CH\(_2\)), 6.85–6.56 (m, 5H, Ar–H), 7.25–7.10 (m, 4H, Ar–H) ppm. \(^13\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\): 158.8, 148.2, 131.4, 129.3, 128.8, 117.5, 114.0, 112.8, 55.2, 47.7 ppm.

**N-(4-N,N-Dimethylbenzyl)aniline** (3c)

IR (neat) v: 3413, 3019, 2931, 2834, 1922, 1600, 1500, 1325, 1239, 1170, 1024, 823, 752, 690 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\): 2.85 (s, 6H), 4.0 (s, br, 1H, NH), 4.32 (s, 2H), 6.43–6.47 (m, 4H), 6.58 (m, 1H), 7.04–6.88 (m, 4H) ppm.[39] \(^13\)C NMR (CDCl\(_3\), 75 MHz) \(\delta\): 147.6, 131.2, 129.6, 127.9, 117.2, 113.5, 46.2, 40.3 ppm.[39]

**N-(4-chlorobenzyl)aniline** (3d)

IR (neat) v: 3671, 3418, 2922, 2851, 1898, 1603, 1508, 1430, 1324, 1271, 1092, 1014, 814, 750, 692 cm\(^{-1}\). \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\): 3.98 (s, br, 1H, NH), 4.25 (s, 2H, CH\(_2\)), 6.73–6.54 (m, 3H, Ar–H), 7.25–7.11 (m, 6H, Ar–H) ppm. \(^13\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\): 147.8, 138.0, 132.8, 129.3, 128.7, 117.8, 112.9, 47.6 ppm.

**N-Benzyl-4-chloroaniline** (3e)

IR (neat) v: 3427, 3028, 2924, 2853, 1952, 1864, 1600, 1502, 1453, 1401, 1321, 1177, 1094, 915, 815, 733, 698, 505 cm\(^{-1}\). \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\): 4.0 (s, br, 1H, NH), 4.25 (s, 2H, CH\(_2\)), 6.49 (d, \(J = 9\) Hz, 2H, Ar–H), 7.06 (d, \(J = 9\) Hz, 2H, Ar–H), 7.35–7.21 (m, 5H, Ar–H) ppm. \(^13\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\): 146.7, 139.0, 129.0, 128.7, 127.4, 127.3, 122.0, 114.0, 48.3 ppm.

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Dibenzylation (3g)
IR (neat) v: 3426, 3423, 3023, 2914, 2852, 1952, 1861, 1598, 1500, 1453, 1404, 1322, 1170, 1094, 915, 815, 732, 668, 507 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\): 1.76 (s, br, NH, 1H), 3.79 (s, 4H, CH\(_2\)), 7.40–7.18 (m, 10H, Ar–H) ppm. \(^13\)C NMR (CDCl\(_3\), 100 MHz) \(\delta\): 140.4, 128.5, 128.3, 127.1, 53.2 ppm.[39]

N-Cyclohexylbenzylamine (3h)
IR (neat) v: 3420, 3023, 2917, 2852, 1952, 1861, 1598, 1500, 1453, 1404, 1322, 1170, 1093, 905, 816, 732, 668, 508 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\): 1.35–1.04 (m, 6H, CH\(_2\)), 1.63–1.59 (m, 1H, NH), 1.75–1.70 (m, 2H, CH\(_2\)), 1.93–1.89 (m, 2H, CH\(_2\)), 2.53–2.43 (m, 1H, CH), 3.80 (s, 2H, CH\(_2\)), 7.33–7.19 (m, 5H) ppm.[40] \(^13\)C NMR (CDCl\(_3\), 75 MHz) \(\delta\): 140.8, 128.2, 128.0, 126.6, 56.2, 51.0, 33.6, 26.2, 25.0 ppm.[40]

1-Benzylpiperidine (3k)
IR (neat) v: 3033, 2983, 2630, 1727, 1379, 1240, 930, 752, 716, 700, 606 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\): 1.45–1.37 (m, 2H, CH\(_2\)), 1.60–1.52 (m, 4H, CH\(_2\)), 2.38–2.34 (m, 4H, CH\(_2\)), 3.46 (s, 2H, CH\(_2\)), 7.32–7.18 (m, 5H, Ar–H) ppm. \(^13\)C NMR (CDCl\(_3\), 100 MHz) \(\delta\): 138.7, 129.3, 128.1, 126.8, 63.9, 54.5, 26.2, 26.0, 24.4 ppm.[39]

4-Benzylmorpholine (3l)
IR (neat) v: 3034, 2986, 2631, 1717, 1377, 1245, 1045, 939, 750, 716, 699, 609 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\): 2.1–2.0 (m, 8H), 4.68 (s, 2H), 7.45–7.28 (m, 5H) ppm. \(^13\)C NMR (CDCl\(_3\), 75 MHz) \(\delta\): 140.5, 129.4, 128.1, 126.9, 66.3, 64.9, 60.4 ppm.

4-Cyclohexylmorpholine (3n)
IR (neat) v: 3422, 3021, 2917, 2852, 1952, 1858, 1596, 1502, 1451, 1400, 1322, 1170, 1084, 900, 809, 731, 648, 500 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\): 1.94–1.09 (m, 10H), 2.25–2.18 (m, 1H), 2.61–2.58 (m, 4H), 3.77–3.75 (m, 4H) ppm.[39] \(^13\)C NMR (CDCl\(_3\), 100 MHz) \(\delta\): 67.5, 63.8, 49.7, 28.9, 26.3, 25.8 ppm.[39]

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N-Benzyl-N-methylaniline (3o)

IR (neat) ν: 3430, 3029, 2926, 2874, 1952, 1850, 1599, 1502, 1451, 1443, 1322, 1178, 1089, 910, 809, 731, 653, 523 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) δ: 2.99 (s, 3H), 4.51 (s, 2H), 6.75–6.68 (m, 3H), 7.32–7.19 (m, 7H) ppm.[39] $^{13}$C NMR (CDCl$_3$, 100 MHz) δ: 138.9, 133.2, 129.7, 128.4, 127.9, 127.1, 121.8, 114.2, 58.1, 41.8 ppm.[39]


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Fig. VB.1. $^1$H NMR spectrum of N-Benzylaniline

Fig. VB.2. $^{13}$C NMR spectrum of N-Benzylaniline

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Fig. VB.3.  *Mass spectrum of N-Benzylationine*

Fig. VB.4.  $^1$H NMR spectrum of 4-Benzylmorpholine

*Reductive amination under microwave irradiation*
Fig. VB.5. $^{13}$C NMR spectrum of 4-Benzylmorpholine

Fig. VB.6. Mass spectrum of 4-Benzylmorpholine