Ionic TEMPO in Ionic Liquids: Specific Promotion of the Aerobic Oxidation of Alcohols

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<th>(Tsunehisa Hirashita, Makoto Nakanishi, Tomoya Uchida, Masakazu Yamamoto, Shuki Araki, Isabel E.C.E. Arends, Roger A. Sheldon)</th>
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Ionic TEMPO in Ionic Liquids: Specific Promotion of the Aerobic Oxidation of Alcohols


Abstract: The main objective of this study was to design a recyclable TEMPO derivative which could be used as a catalyst, in an ionic liquid solvent, for the aerobic oxidation of alcohols using NaNO$_2$ and HCl as co-catalysts. To this end, a TEMPO derivative bearing a quaternary ammonium group, [4-Bu$_3$MeN-TEMPO] [PF$_6$] (1), was prepared. It was subsequently shown that this ionic TEMPO is an efficient catalyst for the aerobic oxidation of a variety of primary and secondary alcohols, exhibiting a synergistic effect with ionic liquid solvents and readily outperforming analogous oxidations in methylene chloride. Moreover, the ionic TEMPO could be recycled five times with no loss of activity.

Introduction

The selective oxidation of alcohols is one of the most important reactions in organic synthesis.[1] There is a growing concern to develop greener oxidation processes that obviate the need for stoichiometric amounts of toxic heavy metals. The 2,2,6,6-tetramethylpiperidinyl-N-oxyl (TEMPO)-catalysed oxidation of alcohols has emerged as a clean and economical oxidation process using bleach or dioxygen as terminal oxidising agents.[2] In general, TEMPO-catalysed oxidations proceed with high chemoselectivity; the over-oxidation of aldehydes to carboxylic acids is very slow, because TEMPO prevents auto-oxidation under aerobic conditions.[3]

Because of its high atom efficiency, oxygen (air) is usually the oxidant of choice and aerobic oxidations are considered one of the greenest oxidation processes; however, they are usually performed with the aid of a catalytic amount of transition-metal compounds. Metal contamination of products causes serious issues in the field of fine chemicals and pharmaceuticals manufacture. The best way to avoid this contamination is to obviate the need for metal catalysts for oxidative processes. Recently, an appealing aerobic oxidation of alcohols catalysed by TEMPO and nitrite or its precursor has been introduced in this field.[4] However, these reactions are normally performed in aqueous biphasic systems using methylene dichloride as organic phase.[5] Hence, from the point of view of green chemistry, the design of a greener protocol for this oxidation is necessary.

Ionic liquids have been used as alternative organic solvents with low vapour pressure, safety issues associated with the flammability of mixtures of oxygen and volatile organic solvents in the gas phase can thus be circumvented. Moreover, ionic liquids have the potential to immobilise catalysts and recycle them for further reaction.[6] Metal-free aerobic oxidation of alcohols with recyclable catalysts immobilised in ionic liquids is a desirable method for the preparation of aldehydes and ketones. The TEMPO-catalysed oxidation in ionic liquids and the recyclability of the ionic liquids in the process have been successfully examined in the literature.[7] Because TEMPO is a rather expensive material, a technique that facilitates its separation from the products and its reuse should be considered. However, extraction of the reaction products from the ionic liquid with an organic solvent results in leaching of TEMPO, which prevents its recycling. In order to overcome this difficulty, TEMPO derivatives containing polar functionalities have been tested.[8a, b, c] Besides their recyclability, the use of ionic liquids as media for the aerobic oxidation of alcohols catalysed by a stable N$_2$O$_2$-oxyl radical, has not been systematically investigated.[7a, 8a, 9] Herein we report an expedient aerobic oxidation of alcohols catalysed by NaNO$_2$/HCl using a suitable ionic liquid and a TEMPO derivative bearing an ammonium moiety. The recyclability of the ionic liquid and catalyst is also discussed.

Results and Discussion

Synthesis of [4-Bu$_3$MeN-TEMPO] [PF$_6$] (1). In order to recycle the catalyst in the ionic liquid, an ammonium tag was introduced into TEMPO (Scheme 1). The ionic TEMPO 1 was prepared by alkylation of 4-aminoo-TEMPO with n-butyl iodide, followed by quaternisation with Me$_3$SiCl. The ionic TEMPO 1 gave [Bu$_3$MeN-TEMPO][PF$_6$] as a pale orange solid in good yield.[10]
Catalytic activity of 1 in aerobic oxidation

We first examined the oxidation of benzyl alcohol (2a) using 3 mol% of ionic TEMPO 1, 5 mol% of NaN₂O₃, and 10 mol% of HCl in [bmim][PF₆]. Benzoic acid was obtained in 99% yield in 1 h (Table 1, entry 1).

Table 1. Oxidation of 2a with 1³¹

<table>
<thead>
<tr>
<th>Entry</th>
<th>Solvent [b]</th>
<th>Time (h)</th>
<th>Yield (%)[a] of Aldehyde [b]</th>
<th>Recovery (%)[a] of 2a [b]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>[bmim][PF₆]</td>
<td>1</td>
<td>99</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>[bmim][NTf₂]</td>
<td>1</td>
<td>53</td>
<td>43</td>
</tr>
<tr>
<td>3</td>
<td>[bmim][NTf₂]</td>
<td>16</td>
<td>99</td>
<td>0</td>
</tr>
<tr>
<td>4</td>
<td>[bmpy][PF₆]</td>
<td>1</td>
<td>52</td>
<td>40</td>
</tr>
<tr>
<td>5</td>
<td>[bmpy][PF₆]</td>
<td>16</td>
<td>99</td>
<td>0</td>
</tr>
<tr>
<td>6²</td>
<td>[bmpy][PF₆]</td>
<td>1</td>
<td>0°</td>
<td>83</td>
</tr>
</tbody>
</table>

[a] Reaction conditions: 2a (0.50 mmol), 1 (3 mol%), NaN₂O₃ (5 mol%), HCl (10 mol%), [bmim][PF₆] (0.50 mmol), 5 h, air, RT, recovery of 2a (75%).
[b] GC yield. Values in parentheses show the recovery of 2a.

In [bmim][NTf₂] a slower oxidation rate was observed than in [bmim][PF₆] (entry 2), and a high yield can be obtained by simply prolonging the reaction time (entry 3). The reaction in [bmpy][PF₆] showed a lower oxidation rate, with a trend similar to that of the reaction performed in [bmim][NTf₂] (entries 4 and 5). In a blank experiment in [bmim][PF₆], benzoic acid was not detected, benzoic acid was formed in 4% yield, and 2a was recovered almost quantitatively. These results demonstrate the importance of a proper choice of the ionic liquid in promoting the oxidation reaction.

Effect of ammonium tag and solvent

In order to confirm the conditions for a faster oxidation, a set of reactions was performed in [bmim][PF₆] and CH₂Cl₂ (Table 2). Compared with the reaction using TEMPO 1 in [bmim][PF₆] oxidation in CH₂Cl₂ was found to be sluggish, affording benzaldehyde in only 49% yield in 10 h (entry 2). In contrast, when TEMPO was used better results were obtained in CH₂Cl₂ (entry 3). Under the conditions reported in the literature⁵⁶, the corresponding aldehyde was obtained in 77% yield, although a longer reaction time (13 h) was needed (entry 4).

Table 2. Solvent and catalyst effect on the oxidation of 2a²¹

<table>
<thead>
<tr>
<th>Entry</th>
<th>Solvent</th>
<th>Time (h)</th>
<th>Yield (%)[a]</th>
<th>TOF (h⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>[bmim][PF₆]</td>
<td>1</td>
<td>99</td>
<td>33</td>
</tr>
<tr>
<td>2</td>
<td>CH₂Cl₂</td>
<td>10</td>
<td>49 (51)</td>
<td>1.6</td>
</tr>
<tr>
<td>3</td>
<td>TEMPO</td>
<td>10</td>
<td>27 (64)</td>
<td>0.9</td>
</tr>
<tr>
<td>4</td>
<td>CH₂Cl₂</td>
<td>13</td>
<td>77 (23)</td>
<td>2.0</td>
</tr>
</tbody>
</table>

[a] Reaction conditions: 2a (0.50 mmol), TEMPO or 1 (0.015 mmol), NaN₂O₃ (0.025 mmol), 1 M HCl (0.050 mmol), solvent (1.5 mL or CH₂Cl₂: 5 mL), air, RT. [b] GC yield. Values in parentheses show the recovery of 2a.

These results clearly indicate that the use of ionic TEMPO 1 in the ionic liquid is crucial for increasing the oxidation rate.

Aerobic oxidation of alcohols with 1

Having established the optimum conditions, the scope and limitations were examined using a series of alcohols (Fig. 1). The results are summarised in Table 3. Benzylc alcohol 2a, 2b, and 3a were easily oxidised to the corresponding aldehydes in high yields (entries 1–3), whereas benzylc alcohol 3b with an electron-withdrawing group was significantly less reactive (entry 4). Allylic alcohol 4 was converted to cinnamaldehyde in good yield (entry 5). Normally, cyclic secondary alcohol 5 resists oxidation because of steric factors; for instance, the TEMPO-
catalysed reaction in CH₂Cl₂ generates cyclohexanone only in 37% conversion and with 91% selectivity after 24 h.[8] In contrast, the present method afforded cyclohexanone in high yields, although a longer reaction time was required (entries 6 and 7). In [bmim][PF₆], aliphatic alcohol 6 was mainly converted into acetal 11 originating from the reaction of the formed aldehyde with [bmim][NTf₂] (entry 8). The formation of acetal in a protic ionic liquid has been previously reported;[11] however, this side reaction can be suppressed by switching the ionic liquid to [bmim][OTf], which affords the corresponding aldehyde in moderate yield (entry 9). Secondary aliphatic alcohols 7 and 8 were converted into the corresponding ketones in excellent yields (entries 10 and 11). Moreover, the oxidation of unsaturated aliphatic alcohol 9 afforded the corresponding aldehyde in moderate yield (entry 12). β-Phenethyl alcohol (10) gave predominantly the corresponding acetal 12 (entry 13).

Using oxygen instead of air in [bmim][NTf₂] under heating was found to accelerate the reaction: phenylacetaldehyde was obtained in moderate yield and formation of acetal 12 was suppressed, presumably by the faster oxidative process (entry 14).

Thus, the optimum ionic liquid for this reaction depends on the alcohol employed. Whereas [bmim][PF₆] was preferable for the oxidation of 2a as shown in Table 1, the use of [bmim][NTf₂] resulted in the selective oxidation of a primary aliphatic alcohol (Table 3, entry 9).

**Aerobic oxidation of octan-2-ol with 1**

We next tested the oxidation of 7, as a representative aliphatic secondary alcohol, in a series of ionic liquids with different anionic counterparts (Table 4). In contrast to the case of primary alcohols, [bmim][PF₆] was superior to [bmim][NTf₂] (entries 1 and 2) and to [bmim][BF₄], which resulted in slow oxidation (entry 3). Ionic liquid [bmim][OTf] was also suitable for the oxidation of 7 (entry 4).

**Table 4. Oxidation of 7 with 1 in ionic liquids**

<table>
<thead>
<tr>
<th>Entry</th>
<th>Solvent</th>
<th>Yield of 2-octanone (%)[b]</th>
<th>Recovery of 7 [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>[bmim][PF₆]</td>
<td>91</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>[bmim][NTf₂]</td>
<td>78</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>[bmim][BF₄]</td>
<td>69</td>
<td>24</td>
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<tr>
<td>4</td>
<td>[bmim][OTf]</td>
<td>91</td>
<td>0</td>
</tr>
</tbody>
</table>

[a] Reaction conditions: 7 (0.50 mmol), 1 (0.015 mmol), NaNO₂ (0.025 mmol), HCl (0.050 mmol), ionic liquid (1.5 mL) under air atmosphere at RT for 5 h. [b] GC yield.

**Recyclability of 1**

Next, the recyclability of the catalytic system was examined in the oxidation of 2a. After the first run, the product was separated from the ionic liquid layer by extraction with diethyl ether. The ionic liquid containing ionic TEMPO 1 was directly reused for the next oxidation. Although addition of new NaNO₂ and HCl was required, no decrease in yield was observed up to five recycles (Table 5).

**Table 5. Recycling of ionic TEMPO 1 and [bmim][PF₆] for the oxidation of 2a**

<table>
<thead>
<tr>
<th>Run</th>
<th>Yield (%)[b]</th>
<th>Recovery of 2a [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>99</td>
<td>0</td>
</tr>
</tbody>
</table>
Stoichiometric oxidation

In order to gain insight into the catalytic role of 1, oxidation of 2a using stoichiometric amount of oxoammonium 1\( ^* \) was tested. Oxoammonium 1\( ^* \) was prepared according to the literature procedure described for [TEMPO]\( ^* \)[BF\(_4\)].\(^{[12]} \) After exposure of 1 to HPF\(_6\),\(^{[13]} \) the reaction mixture was treated with NaOCl. The purity of the resulting oxoammonium salt was estimated by oxidation of an excess amount of benzyl alcohol. TEMPO\(^*\) obtained by this procedure showed a good purity (98%), whereas the oxoammonium derived from 1 was only 46% pure (Scheme 3). However, a satisfactory result was obtained using a double amount of reagents (88% purity of 1\( ^* \)).

![Scheme 3. Preparation of oxoammonium salts.](image)

Table 6. Stoichiometric oxidation of 2a with TEMPO\(^*\) or 1\(^*\)([13])

<table>
<thead>
<tr>
<th>Entry</th>
<th>Oxidant</th>
<th>Solvent</th>
<th>Time (h)</th>
<th>Yield of aldehyde (%)</th>
<th>Recovery of 2a (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1(^*)</td>
<td>[bmim][PF(_6)]</td>
<td>0.25</td>
<td>96</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>1(^*)</td>
<td>CH(_3)CN</td>
<td>0.25</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>1(^*)</td>
<td>CH(_3)Cl</td>
<td>1.5</td>
<td>64</td>
<td>36</td>
</tr>
<tr>
<td>4</td>
<td>TEMPO(^*)</td>
<td>[bmim][PF(_6)]</td>
<td>1.5</td>
<td>23</td>
<td>66</td>
</tr>
<tr>
<td>5</td>
<td>TEMPO(^*)</td>
<td>CH(_3)CN</td>
<td>1.5</td>
<td>70</td>
<td>24</td>
</tr>
<tr>
<td>6</td>
<td>TEMPO(^*)</td>
<td>CH(_3)Cl</td>
<td>1.5</td>
<td>92</td>
<td>6</td>
</tr>
</tbody>
</table>

[a] Reaction conditions: 2a (0.09 mmol), TEMPO\(^*\) or 1\(^*\) (0.10 mmol) in solvent (1.0 mL) at RT. [b] Determined by GC.

Comparison of the results in Tables 2 and 6 reveals a similar tendency in the stoichiometric and catalytic oxidations. The reaction with 1\(^*\) in CH\(_3\)CN and [bmim][PF\(_6\)] proceeded faster than that in CH\(_2\)Cl\(_2\) (entries 1–3), whereas TEMPO\(^*\) was most efficient in CH\(_2\)Cl\(_2\) (entries 4–6). It should be noted that 1\(^*\) was not completely dissolved in CH\(_2\)Cl\(_2\), and therefore the above results may not accurately reflect the reaction rate of 1\(^*\) in CH\(_2\)Cl\(_2\).

Comparison of TEMPO and ionic TEMPO (1) in ionic liquids

For the catalytic and stoichiometric oxidations, CH\(_2\)CN, which is typically used for TEMPO-catalysed reactions, was a superior solvent to [bmim][PF\(_6\)]. The oxidation in acetonitrile also yielded better results than those obtained in [bmim][PF\(_6\)]. This is in good agreement with the literature reports,\(^{[9a]} \) which ascribe this tendency to the different viscosities of molecular solvents and ionic liquids. In contrast, TEMPO\(^*\)-mediated oxidations in an alternative ionic liquid, N-buty1-N-methyl pyrrolidinium bis(trifluoromethylsulfonyl)imide, proceeded faster than those in acetonitrile,\(^{[8c]} \) suggesting that the reaction rate of the TEMPO\(^*\)-mediated oxidation is strongly dependent on the ionic liquid employed. On the other hand, the results related to the use of 1 were found to be quite different. The reactions in [bmim][PF\(_6\)] proceeded at a clearly faster rate than those performed in CH\(_2\)Cl\(_2\), and approximately the same results were obtained in acetonitrile. This trend can be rationalized by assuming that more polar solvents preferably promote the formation and reaction of the highly active oxoammonium 1\(^*\) containing both oxoammonium and ammonium groups,\(^{[14]} \) thus compensating for the high viscosity of the ionic liquid. As compared with the ionic liquid, acetonitrile served as a good solvent giving the same level of conversion. However, [bmim][PF\(_6\)] is superior to acetonitrile in separating the products from the reaction mixture while keeping the catalyst in the solvent to be reused in the next reaction.

Conclusions

TEMPO derivative 1 proved to be an effective mediator for the aerobic oxidation of alcohols in ionic liquids. This study clearly demonstrates the importance of careful screening of ionic liquids with respect to the selectivity as well as the acceleration. In addition, a strong synergistic effect between ionic-TEMPO 1 and ionic liquids became apparent. The use of 1 and an ionic liquid plays a key role in increasing the oxidation rate. The ammonium tag on 1 is responsible not only for recycling the N-oxo radical in the ionic liquid phase but also for enhancing the oxidation rate.
Experimental Section

General Methods

IR spectra were recorded on a JASCO IRA-102 spectrophotometer. 1H NMR spectra were obtained in CDCl₃ or CD₂CN using a Varian Mercury 300 spectrometer (300 MHz) with Me₄Si as internal standard; J-values are given in Hz. GC analyses were performed on a SHIMADZU GC-14B and GC-2014 fitted with a capillary column and a flame ionization detector. Elemental analyses were carried out with a Perkin Elmer 2400II analyzer. Melting points were measured on a Yanaco MP5533 and are uncorrected. Ionic liquids were purchased from Kanto Chemical and were used as received. All chemicals were commercially available and distilled before use.

Experimental Procedures

Preparation of 4-Bu₅N-TEMPO: A mixture of 4-amino-TEMPO (1.62 g, 9.45 mmol), n-butyl iodide (4.5 mL, 39.6 mmol), and K₂CO₃ (8.23 g, 59.5 mmol) in acetonitrile (50 mL) was refluxed for 23 h. The product was diluted with diethyl ether (150 mL), washed with water (40 mL × 3) and brine, and dried over Na₂SO₄. The solvent was removed under reduced pressure to give 4-Bu₅N-TEMPO (2.38 g, 89%).

1H NMR (300 MHz, CDCl₃): δ = 0.86–0.90 (m, 9H), 1.2–1.4 (m, 30H), 1.54–1.59 (m, 6H), 3.39–3.44 (m, 2H), 3.53–3.57 (m, 2H), 4.46 ppm (t, J = 5.7 Hz, 1H); 13C NMR (75 MHz, CDCl₃): δ = 49.1, 66.2 ppm.

Preparation of 4-Bu₅N-TEMPO: 4-Bu₅N-TEMPO (2.24 g, 7.92 mmol) was treated with MeI (2.5 mL, 40 mmol) in acetonitrile (40 mL) and heated at reflux for 3 h. The complete consumption of 4-Bu₅N-TEMPO, as monitored by TLC, the solvent was removed under reduced pressure affording [4-Bu₅MeN-TEMPO] (3.23 g, 96%).

Preparation of ionic TEMPO 1: A mixture of benzyl alcohol (10.2 mg, 0.094 mmol) and ionic TEMPO (2.24 g, 7.92 mmol) was added over 3 minutes at 0 °C, and the mixture was stirred for 40 minu. After stirring for 40 minutes, NaOCl (1.84 M, 136 µL, 0.25 mmol) was added over 3 minutes at 0 °C, and the mixture was stirred for further 1 hour. The product was filtered and washed with cold aqueous 5% NaHCO₃ water, and cold diethyl ether. After drying, [TEPOM⁺][PF₆⁻] was obtained as bright yellow solid (88 mg).

Preparation and titration of TEMPO⁺[PF₆⁻]‡

To a mixture of TEMPO (78.6 mg, 0.50 mmol) and water (0.2 mL), HPF₆ (65 wt%, 115 µL, 0.50 mmol) was slowly added over a period of 3 minutes at room temperature. After stirring for 40 minutes, NaOCl (1.84 M, 136 µL, 0.25 mmol) was added over 3 minutes at 0 °C, and the mixture was stirred for further 1 hour. The product was filtered and washed with cold aqueous 5% NaHCO₃ water, and cold diethyl ether. After drying, [1][PF₆⁻] was obtained as a yellow solid (118 mg). A mixture of benzyl alcohol (10.4 µL, 0.10 mmol) and [1][PF₆⁻] (29.5 mg, 0.050 mmol) in CH₂Cl₂ (4 mL) was stirred at room temperature for 17 h. The product was extracted with ether. The yield of benzyldiazcyle (98%) based on [TEPOM⁺][PF₆⁻] was determined by GC using hexadecane as an internal standard.

Preparation of ionic TEMPO 1: A mixture of benzyl alcohol (111 mg, 0.25 mmol) and water (0.4 mL), HPF₆ (65 wt%, 115 µL, 0.50 mmol) was slowly added over a period of 3 minutes at room temperature. After stirring for 40 minutes, NaOCl (1.84 M, 136 µL, 0.25 mmol) was added over 3 minutes at 0 °C, and the mixture was stirred for further 1 hour. The product was filtered and washed with cold aqueous 5% NaHCO₃ water, and cold diethyl ether. After drying, [1][PF₆⁻] was obtained as a yellow solid (118 mg). A mixture of benzyl alcohol (10.4 µL, 0.10 mmol) and [1][PF₆⁻] (29.5 mg, 0.050 mmol) in CH₂Cl₂ (4 mL) was stirred at room temperature for 17 h. The product was extracted with ether. The yield of benzyldiazcyle (98%) based on [1][PF₆⁻] was determined by GC using hexadecane as an internal standard.

Stoichiometric oxidation of benzyl alcohol by ionic TEMPO 1* (Table 6, entry 1)

A mixture of benzyl alcohol (10.2 mg, 0.094 mmol) and ionic TEMPO 1* (88% purity, 67 mg, 0.10 mmol) in [bmim][PF₆] (1.0 mL) was stirred at room temperature for 15 minutes. The product was extracted with diethyl ether (2 mL × 3). The yield was determined by GC analysis using hexadecane as an internal standard.
Acknowledgements

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Keywords: Alcohols • Ionic liquids • Oxidation • Oxygen • Radicals


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