



Preliminary communication/Communication

# Efficient and convenient oxidation of alcohols to aldehydes and ketones with molecular oxygen mediated by $\text{In}(\text{NO}_3)_3$ in ionic liquid $[\text{C}_{12}\text{mim}][\text{FeBr}_4]$

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## ABSTRACT

A simple, efficient, and ecofriendly procedure for the aerobic oxidation of alcohols to aldehydes and ketones in the presence of  $\text{In}(\text{NO}_3)_3/[\text{C}_{12}\text{mim}][\text{FeBr}_4]$  in aqueous media has been developed. The oxidation reactions afford the target products in good to high yields and no over-oxidation was observed. The products can be separated by a simple extraction with dichloromethane, and the system can be recycled and reused without loss of activity.

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## 1. Introduction

Aldehydes and ketones are important classes of chemicals that have been used extensively as synthetic intermediates in the preparation of a variety of fine or special chemicals, such as drugs, vitamins, fragrances, etc. [1]. Up to now, many publications in the open literature have been found related to the synthesis of these types of compounds, and a well-known method for such a synthesis is the oxidation of alcohols [2]. Traditional methods for performing such a transformation generally involve the use of stoichiometric amounts of strong oxidizing reagents (e.g.,  $\text{MnO}_2$ ,  $\text{SeO}_2$ ,  $\text{CrO}_3$ , and  $\text{RuO}_4$ ) [2–4], and suffer from considerable drawbacks, such as high cost, low yield, harsh or delicate reaction conditions, and a large amount of waste by-products. Oxidation with ceric ammonium nitrate [5],  $\text{H}_5\text{IO}_6$  [6], diaziridinone [7], *tert*-butyl hydroperoxide [8],

trichloroisocyanuric acid [9,10],  $\text{H}_2\text{O}_2$  [11–13], and other reagents [14–16] have also been developed for this conversion. However, some of the procedures are often associated with one or more disadvantages, such as long reaction times, high temperatures, difficulties in work-up, use of expensive, toxic and moisture-sensitive reagents, and difficulties in recycling the catalyst.

Molecular oxygen has recently been utilized as an attractive oxidant for the oxidation of alcohols for the production of these compounds due to cheap price and environmental friendly nature. Therefore, molecular oxygen-based protocols are powerful methods for oxidation of alcohols and attract a great attention, which make the reactions of choice in large- and industrial-scale applications. A variety of catalytic systems, such as nanopalladium [17],  $\text{Pd}(\text{OAc})_2/\text{DMSO}$  [18],  $\text{Ru}(\text{TPFP})\text{(CO)}$  [19],  $\text{PI Ru/TEMPO}$  [20],  $\text{CeO}_3/\text{RuCl}_3$  [21]  $\text{VOPO}_4/\text{TEMPO}$  [22],  $\text{VO}(\text{acac})_2/\text{DABCO}$  [23], [copper(II)-(N ligand)<sub>n</sub>]/TEMPO [24], gold [25],  $\text{OsHCl}(\text{CO})(\text{P}i\text{Pr}_3)_2$  [26], and other systems [27–29], have been developed for this conversion. However, these procedures are invariably associated with one

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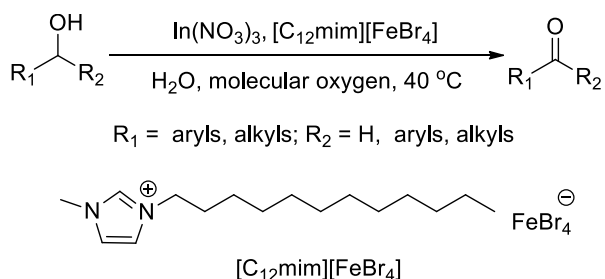
or more disadvantages, such as long reaction times, expensive metals, low yields, use of toxic compounds, difficulties in work-up, harmfulness toward the environment, etc. Consequently, the search for new and environmentally benign synthetic methodologies for the oxidation of alcohols that address these drawbacks remains of value and interest.

Ionic liquids (ILs) have attracted considerable attention during the past few years due, in some cases, to their favourable properties, such as very low vapour pressure, wide liquid temperature range, good ionic conductivity, excellent electrochemical properties, and strong ability to dissolve many chemicals [30–33]. Therefore, ILs have recently gained recognition from scholars from various fields, such as analytical and separation science [34], chemical synthesis [35,36], biocatalytic transformations [37], electrochemistry [38], and catalysis [39–41]. In continuation of our interest in exploring green oxidation reactions in ionic liquids, we report herein a new, efficient and environmentally friendly protocol for the selective aerobic oxidation of alcohols to the aldehydes and ketones mediated by indium nitrate ( $\text{In}(\text{NO}_3)_3$ ) in ionic liquid 1-dodecyl-3-methylimidazolium iron bromide ( $[\text{C}_{12}\text{mim}][\text{FeBr}_4]$ ) under mild conditions (Scheme 1). Furthermore, we demonstrate that the catalytic system can be recycled and reused without any significant loss of activity.

## 2. Experimental

### 2.1. General

All the chemicals were from commercial sources without any pre-treatment. All reagents were of analytical grade. The ionic liquids were synthesized according to the literature procedure [33].  $^1\text{H}$  NMR spectra were recorded on a Bruker 500-MHz spectrometer using  $\text{CDCl}_3$  as the solvent with tetramethylsilane (TMS) as an internal standard. High-performance liquid chromatography (HPLC) experiments were performed on a liquid chromatograph (Dionex Softron GmbH, America), consisting of a pump (P680) and ultraviolet-visible light detector (UVD) system (170U). The experiments were performed on Diacoverly C18 column,  $\varnothing 4.6 \times 250$  mm. Elemental analysis was performed on a Vario EL III instrument (Elementar Analysensysteme GmbH, Germany).



Scheme 1. Aerobic oxidation of alcohols to aldehydes and ketones.

### 2.2. General procedure for oxidation reactions

All the three-necked flasks were loaded with alcohol (0.1 mol),  $\text{In}(\text{NO}_3)_3$  (0.3 mol),  $[\text{C}_{12}\text{mim}][\text{FeBr}_4]$  (30 mL), and  $\text{H}_2\text{O}$  (10 mL).  $\text{O}_2$  gas was bubbled into the flask at the flow rate of 60 mL/min. The reaction mixture was stirred at  $40^\circ\text{C}$  for an appropriate time (Table 3), the reaction progress was monitored by HPLC. The mixture was then cooled to room temperature and the organic phase was extracted with dichloromethane ( $3 \times 20$  mL). The dichloromethane solution was washed with 5%  $\text{NaHCO}_3$  ( $2 \times 20$  mL) and water ( $3 \times 20$  mL), and then dried over anhydrous  $\text{Na}_2\text{SO}_4$ . The solvent was removed and the residue was distilled under vacuum to give the desired pure product. The rest of the ionic liquid and  $\text{In}(\text{NO}_3)_3$  was recovered. Fresh substrates were then recharged to the recovered catalytic system and then recycled under identical reaction conditions. The target substrates were characterized by elemental analysis,  $^1\text{H}$  NMR spectra or compared with their authentic samples. Spectroscopic data for selected products is as follows.

#### 2.2.1. 4-Tert-butylbenzaldehyde

Colourless oil.  $^1\text{H}$  NMR:  $\delta = 1.31$  (s,  $\text{CH}_3$ , 9H), 7.51–7.72 (m, Ar–H, 4H), 9.94 (s, CHO, 1H). Anal. calcd. for  $\text{C}_{11}\text{H}_{14}\text{O}$ : C, 81.39; H, 8.68; O, 9.84. Found: C, 81.44; H, 8.70; O, 9.86 (Table 3, entry 4).

#### 2.2.2. 2,4,6-Trimethylbenzaldehyde

Colourless oil.  $^1\text{H}$  NMR:  $\delta = 2.15$  (s,  $\text{CH}_3$ , 3H), 2.39 (s,  $\text{CH}_3$ , 6H), 7.21 (s, Ar–H, 2H), 9.89 (s, CHO, 1H). Anal. calcd. for  $\text{C}_{10}\text{H}_{12}\text{O}$ : C, 81.03; H, 8.14; O, 10.76. Found: C, 81.02; H, 8.16; O, 10.78 (Table 3, entry 5).

#### 2.2.3. 4-Acetylbenzaldehyde

White solid, mp:  $35\text{--}37^\circ\text{C}$ .  $^1\text{H}$  NMR:  $\delta = 2.63$  (s,  $\text{CH}_3$ , 3H), 7.91–7.96 (m, Ar–H, 2H), 8.17–8.22 (m, Ar–H, 2H), 9.96 (s, CHO, 1H). Anal. calcd. for  $\text{C}_9\text{H}_8\text{O}_2$ : C, 72.91; H, 5.42; O, 21.57. Found: C, 72.96; H, 5.44; O, 21.60 (Table 3, entry 6).

#### 2.2.4. trans-Cinnamaldehyde

Colourless oil.  $^1\text{H}$  NMR:  $\delta = 6.65$  (d,  $J = 16.5$  Hz, CH, 1H), 6.67 (dd,  $J = 6.5, 16.5$  Hz, CH, 1H), 7.34–7.47 (m, Ar–H, 5H), 9.69 (d,  $J = 6.5$  Hz, CHO, 1H). Anal. calcd. for  $\text{C}_9\text{H}_8\text{O}$ : C, 81.79; H, 6.10; O, 12.11. Found: C, 81.79; H, 6.10; O, 12.11 (Table 3, entry 10).

#### 2.2.5. 3,7-Dimethyl-2,6-octadienal

Colourless liquid.  $^1\text{H}$  NMR:  $\delta = 1.59$  (s,  $\text{CH}_3$ , 3H), 1.93 (s,  $\text{CH}_3$ , 3H), 2.21 (s,  $\text{CH}_3$ , 3H), 2.58 (m,  $\text{CH}_2\text{CH}_2$ , 4H), 5.07 (s, CH, 1H), 5.87 (d,  $J = 7.0$  Hz, CH, 1H), 9.85 (d,  $J = 7.0$  Hz, CHO, 1H). Anal. calcd. for  $\text{C}_{10}\text{H}_{16}\text{O}$ : C, 78.87; H, 10.59; O, 10.52. Found: C, 78.90; H, 10.59; O, 10.51 (Table 3, entry 11).

## 3. Results and discussion

A preliminary survey of the reaction conditions (Table 1) used benzyl alcohol (0.1 mol) as the model substrate in the presence of ionic liquid  $[\text{C}_{12}\text{mim}][\text{FeBr}_4]$  and  $\text{In}(\text{NO}_3)_3$ . In the absence of ionic liquid and  $\text{In}(\text{NO}_3)_3$ ,

**Table 1**

[C<sub>12</sub>mim][FeBr<sub>4</sub>]/In(NO<sub>3</sub>)<sub>3</sub> catalysed the oxidation of benzyl alcohol with molecular oxygen<sup>a</sup>.

Entry	[C <sub>12</sub> mim][FeBr <sub>4</sub> ] (mL)	In(NO <sub>3</sub> ) <sub>3</sub> (equiv.)	Time (h)	Yield (%) <sup>b</sup>
1	–	–	24	0
2	10	–	12	28
3	–	1.0	12	23
4	20	1.0	8	52
5	28	1.0	8	64
6	30	1.0	8	67
7	32	1.0	8	67
8	30	2.0	8	81
9	30	2.8	6	90
10	30	3.0	6	92
11	30	3.2	6	92

<sup>a</sup> The reactions were carried out with benzyl alcohol (0.1 mol), In(NO<sub>3</sub>)<sub>3</sub> (0.3 mol), [C<sub>12</sub>mim][FeBr<sub>4</sub>] (30 mL), and H<sub>2</sub>O (10 mL) with the bubbling of O<sub>2</sub> gas (60 mL/min) at 40 °C.

<sup>b</sup> Isolated yield of benzaldehyde.

no product was detected (Table 1, entry 1). When the reaction was performed in the presence of ionic liquid [C<sub>12</sub>mim][FeBr<sub>4</sub>] (10 mL) or catalyst In(NO<sub>3</sub>)<sub>3</sub> (1.0 equiv), the product could be obtained in a higher yield 28% (Table 1, entry 2) or 23% in a shorter time (12 h) (Table 1, entry 3) under the same conditions. The yield can be greatly improved with the combined use of [C<sub>12</sub>mim][FeBr<sub>4</sub>] and In(NO<sub>3</sub>)<sub>3</sub> (Table 1, entries 4–11), and it reached its maximum value (92%) within a shorter time (6 h) when the addition of [C<sub>12</sub>mim][FeBr<sub>4</sub>] (30 mL) and In(NO<sub>3</sub>)<sub>3</sub> (3.0 equiv.) (Table 1, entry 10). However, upon further addition of [C<sub>12</sub>mim][FeBr<sub>4</sub>] (Table 1, entry 7) or In(NO<sub>3</sub>)<sub>3</sub> (Table 1, entry 11) under the same conditions, the yield was not enhanced significantly. These experiments revealed that In(NO<sub>3</sub>)<sub>3</sub> and ionic liquid [C<sub>12</sub>mim][FeBr<sub>4</sub>] are crucial for the oxidation, and the lack of any component can lead to a much lower yield. Therefore, the optimal reaction conditions were observed in Table 1, entry 10.

The effects of different ionic liquids, such as [C<sub>8</sub>mim][FeBr<sub>4</sub>], [C<sub>12</sub>mim][FeBr<sub>4</sub>], [C<sub>12</sub>mim][FeCl<sub>4</sub>], [C<sub>12</sub>mim][OTf], [C<sub>12</sub>mim][BF<sub>4</sub>], and [C<sub>12</sub>mim]Br, were tested with In(NO<sub>3</sub>)<sub>3</sub> as a catalyst in the reaction (Table 2, entries 1–6), and it was observed that [C<sub>12</sub>mim][FeBr<sub>4</sub>] demonstrated the best performance (Table 2, entry 2). The different catalytic effects of ILs may be attributed to their different abilities to stabilize and dissolve O<sub>2</sub>, the catalyst and the substrate. Under our reaction conditions, O<sub>2</sub> may be more soluble in [C<sub>12</sub>mim][FeBr<sub>4</sub>], leading to a higher effective concentration of the oxidant, which increases the encounter probabilities between the substrate and the reactive species, and so the higher rate and yield of the reaction were observed. Furthermore, besides In(NO<sub>3</sub>)<sub>3</sub>, other types of catalysts were also tested in this model reaction (Table 2, entries 7–11). The results showed that In(NO<sub>3</sub>)<sub>3</sub> demonstrated the best performance. In addition, no over-oxidized product (benzoic acid) was detected by <sup>1</sup>H NMR analysis of the crude reaction mixtures in all the cases. Therefore, the combination of [C<sub>12</sub>mim][FeBr<sub>4</sub>] and In(NO<sub>3</sub>)<sub>3</sub> was chosen as the optimal conditions for further exploration.

**Table 2**

Optimization of the conditions for oxidation of benzyl alcohol with molecular oxygen<sup>a</sup>.

Entry	Ionic liquid	Catalyst	Time (h)	Yield (%) <sup>b</sup>
1	[C <sub>8</sub> mim][FeBr <sub>4</sub> ]	In(NO <sub>3</sub> ) <sub>3</sub>	6	89
2	[C <sub>12</sub> mim][FeBr <sub>4</sub> ]	In(NO <sub>3</sub> ) <sub>3</sub>	6	92
3	[C <sub>12</sub> mim][FeCl <sub>4</sub> ]	In(NO <sub>3</sub> ) <sub>3</sub>	6	85
4	[C <sub>12</sub> mim][OTf]	In(NO <sub>3</sub> ) <sub>3</sub>	6	41
5	[C <sub>12</sub> mim][BF <sub>4</sub> ]	In(NO <sub>3</sub> ) <sub>3</sub>	6	56
6	[C <sub>12</sub> mim]Br	In(NO <sub>3</sub> ) <sub>3</sub>	6	78
7	[C <sub>12</sub> mim][FeBr <sub>4</sub> ]	Cu(NO <sub>3</sub> ) <sub>2</sub>	6	66
8	[C <sub>12</sub> mim][FeBr <sub>4</sub> ]	Fe(NO <sub>3</sub> ) <sub>3</sub>	6	70
9	[C <sub>12</sub> mim][FeBr <sub>4</sub> ]	AgNO <sub>3</sub>	6	78
10	[C <sub>12</sub> mim][FeBr <sub>4</sub> ]	MnO <sub>2</sub>	8	45
11	[C <sub>12</sub> mim][FeBr <sub>4</sub> ]	Ba(NO <sub>3</sub> ) <sub>2</sub>	6	61

<sup>a</sup> The reactions were carried out with benzyl alcohol (0.1 mol), catalyst (0.3 mol), IL (30 mL), and H<sub>2</sub>O (10 mL) with the bubbling of O<sub>2</sub> gas (60 mL/min) at 40 °C.

<sup>b</sup> Isolated yield of benzaldehyde.

In addition, the catalytic system could be typically recovered and reused for subsequent reactions with no appreciable decrease in yields and reaction rates (Fig. 1). The recycling process involved the removal of the upper layer of product, by a simple extraction with organic solvent. Fresh substrates were then recharged to the under-layer of catalytic system and the mixture was heated to react once again.

With these results in hand, the catalytic system was then applied to various alcohols as summarized in Table 3. It is clear that various types of benzylic and allylic alcohols, both primary and secondary, have been successfully oxidized to the corresponding aldehydes and ketones in good to high conversions and yields (Table 3, entries 1–11), whereas the aliphatic alcohols were less reactive, and the reactions proceeded more slowly, and even under more drastic reaction conditions, the reactions were still incomplete (Table 3, entries 12 and 13). Surprisingly, the oxidation of primary alcohols to the corresponding carbonyl compounds is faster and more efficient than that of secondary alcohols (Table 3, entries 8 and 9). In addition, it was also observed that the electronic nature of the substituents on the aromatic ring has some impact on the reaction rate. Benzylic alcohols, especially those with electron-donating substituents (Table 3, entries 2–5), were more reactive than those with electron-withdrawing substituents (Table 3, entries 6 and 7), providing excellent yields.

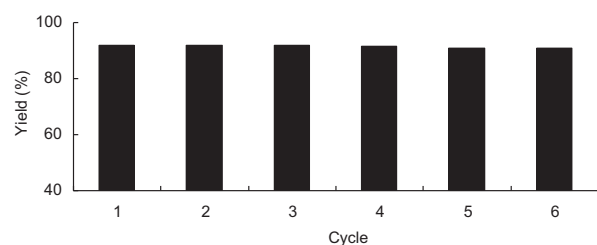
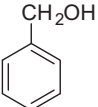
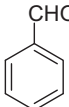
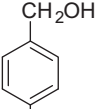
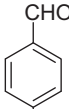
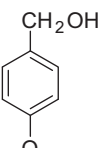
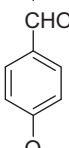
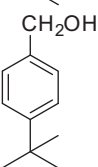
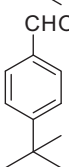
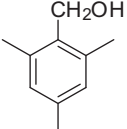
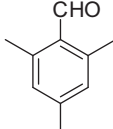
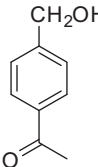
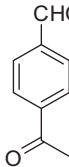
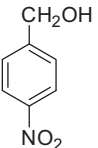
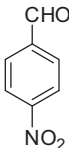
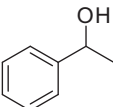
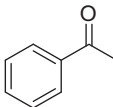
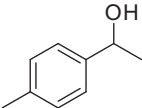
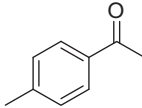
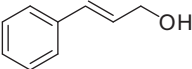
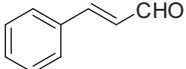
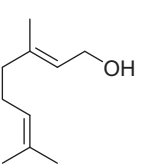
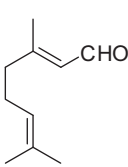
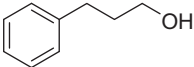
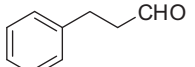
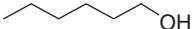
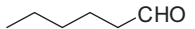


Fig. 1. Repeated reactions using recovered In(NO<sub>3</sub>)<sub>3</sub>.

**Table 3**  
Oxidation of alcohols to aldehydes and ketones<sup>a</sup>.

Entry	Substrate	Product	Time (h)	Conversion (%) <sup>b</sup>	Yield (%) <sup>c</sup>
1			6	100	92
2			6	100	94
3			6	100	95
4			6	100	93
5			6	100	96
6			10	96	89
7			10	95	86
8			8	100	90
9			8	100	91
10			6	100	95
11			6	100	93

**Table 3** (Continued)

Entry	Substrate	Product	Time (h)	Conversion (%) <sup>b</sup>	Yield (%) <sup>c</sup>
12			24	84	75 <sup>d</sup>
13			24	81	67 <sup>d</sup>

<sup>a</sup> Unless otherwise specified, the reactions were carried out with alcohol (0.1 mol),  $\text{In}(\text{NO}_3)_3$  (0.3 mol),  $[\text{C}_{12}\text{mim}][\text{FeBr}_4]$  (30 mL), and  $\text{H}_2\text{O}$  (10 mL) with  $\text{O}_2$  gas bubbling (60 mL/min) at 40 °C.

<sup>b</sup> Isolated conversion.

<sup>c</sup> Isolated yield.

<sup>d</sup> The reaction was carried out at 60 °C.

#### 4. Conclusions

In conclusion, a new efficient oxidation reaction system using  $[\text{C}_{12}\text{mim}][\text{FeBr}_4]$  in combination with  $\text{In}(\text{NO}_3)_3$  has been developed, which is capable of converting alcohols into the corresponding aldehydes and ketones in good to excellent isolated yield. Mild reaction conditions, simplicity of operation, high yields, stability, easy isolation of products, and the excellent recyclability of the catalytic system are the attractive features of this methodology. The scope, definition of the mechanism and synthetic application of this reaction are currently under study in our laboratory.

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