Another application of newly prepared Brønsted-acidic ionic liquids as highly efficient reusable catalysts for neat synthesis of amidoalkyl naphthols

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Another application of newly prepared Brønsted-acidic ionic liquids as highly efficient reusable catalysts for neat synthesis of amidoalkyl naphthols

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Abstract: In this work, two newly prepared Brønsted-acidic ionic liquids, [MPyrSO3H]Cl (IL1) and [MMorSO3H]Cl (IL2), were efficiently used as catalysts for the synthesis of amidoalkyl naphthols through the one-pot, three-component reaction of β-naphthol, aryl aldehydes, and acetamide under neat conditions. High activity of the catalysts, excellent yields, short reaction times, simple procedure with an easy work-up, and the absence of any volatile and hazardous organic solvents are some advantages of the present methodology. Moreover, the catalysts are simply prepared and can be recovered conveniently and reused such that considerable catalytic activity can still be achieved after the fifth run.

Keywords: Brønsted-acidic ionic liquids; amidoalkyl naphthols; solvent-free conditions

1. Introduction
A major challenge in modern chemistry is the design of highly efficient chemical reaction sequences that provide maximum structural complexity with a minimum number of synthetic steps in short reaction times (Dömling, 2006; Schreiber, 2000). Multicomponent reactions (MCRs) have gained considerable attention as a powerful method in organic synthesis and medicinal chemistry because they involve simultaneous reaction of more than two starting materials to yield a single product through one-pot reaction (Gore & Rajput, 2013; Slobbe, Ruijter, & Orru, 2012; Tavakoli-Hoseini &
High atom economy, good selectivity, time and energy saving, low cost, minimum waste production, and short reaction time make MCRs suitable for the synthesis of complex molecules with potential biological activity (Chebanov & Desenko, 2012; Manjappa, Peng, Jhang, & Yang, 2016; Zang, Zhang, Zang, & Cheng, 2010). On the other hand, the nature of the catalyst plays a crucial role in the determination of the product and selectivity (Khan, Khan, & Bannuru, 2010; Mirzaei & Davoodnia, 2012; Shaterian & Mohammadnia, 2012). Therefore, development of inexpensive, mild, and reusable catalysts for MCRs such as the synthesis of amidoalkyl naphthols remains of interest to the synthetic organic chemists. It has been reported that amidoalkyl naphthols can convert to important biologically active aminoalkyl naphthol derivatives by amide hydrolysis. Later compounds have been evaluated for the hypotensive and bradycardiac effects (Dingermann, Steinhilber, & Folkers, 2004; Shen, Tsai, & Chen, 1999). Amidoalkyl naphthols are generally synthesized via the three-component reaction of β-naphthol, an aldehyde, and an amide in the presence of various catalysts, such as Sb(OAc)₃ (Hakimi, 2016), zirconocene dichloride (Cp₂ZrCl₂) (Khanapure, Jagadale, Salunkhe, & Rashinkar, 2016), ZrOCl₂·8H₂O (Sheik Mansoor, Aswin, Logaiya, & Sudhan, 2016), nano Al₂O₃ (Kiasat, Hemat-Alian, & Saghanezhad, 2016), carbon-based solid acid (Davoodnia, Mahjoobin, & Tavakoli-Hoseini, 2014), nano-sulfated zirconia (Zali & Shokrolahi, 2012; Sheik Mansoor, Aswin, Logaiya, & Sudhan, 2016), K₅CoW₁₂O₴₀·3H₂O (Nagarapu, Baseeruddin, Apuri, & Kantevari, 2007), copper p-toluensulfonate (Wang & Liang, 2011), Al(H₃PO₄)₃ (Shaterian, Amirzadeh, Khorami, & Ghashang, 2008), Yb(OTf)₃ in [bmim][BF₄] (Kumar, Rao, Ahmad, & Khungar, 2009), and nano silica phosphoric acid (Bamoniri, Mirjalili, & Nazemian, 2014). Although each of these individual methods has its own merits, many suffer from limitations such as long reaction times, unsatisfactory yields, and the use of relatively expensive catalysts. Thus, the exploration of novel methodologies using new efficient and reusable catalysts is still ongoing.

In recent years, ionic Liquids (ILs) have attracted rising interest as eco-friendly solvents, catalysts and reagents in organic transformations due to their advantageous properties, such as non-flammability, negligible vapor pressure, high thermal and chemical stability, and ability to dissolve a wide range of materials (Chowdhury, Mohan, & Scott, 2007; Olivier-Bourbigou, Magna, & Morvan, 2010; Pârvulescu & Hardacre, 2007). ILs are miscible with materials having very wide range of polarities and are simultaneously able to dissolve a wide range of organic, inorganic and organometallic substances. These features offer numerous opportunities for the improvement of organic reactions using ILs as solvents and catalysts. Moreover, their ionic character enhances the reaction rates to a great extent in many reactions. Among them, Brønsted acidic ILs, especially the SO₃H-functionalized ones, have designed as environmentally friendly catalysts to replace the traditional mineral liquid acids like sulfuric acid and hydrochloric acid in chemical processes (Greaves & Drummond, 2008; Qiu et al., 2016; Shirole, Kadnor, Tambe, & Shelke, 2017; Vafaeezadeh & Alinezhad, 2016; Zolfigol, Khazaie, Moosavi-Zare, & Zare, 2010).

Considering the unique properties of Brønsted-acidic ILs, recently, we have synthesized two sulfonic acid functionalized ILs, including 1-methyl-1-sulfonic acid pyrrolidinium chloride [MPyrrSO₃H][Cl] (IL₁) and 4-methyl-4-sulfonic acid morpholinium chloride [MMorSO₃H][Cl] (IL₂) (Figure 1), and successfully applied them as highly efficient catalysts in the synthesis of 1,8-dioxooctahydroxanthenes (Dehghan, Davoodnia, Bozorgmehr, & Bamoharram, 2016). These findings encouraged us to explore other applications of these ILs in the synthesis of organic compounds. Therefore, in line with our interest on the development of convenient methods using reusable catalysts (Davoodnia, 2011; Davoodnia, Allameh, Fazi, & Tavakoli-Hoseini, 2011; Davoodnia, Basafa, & Tavakoli-Hoseini, 2016; Davoodnia, Khajastehnezad, Bakavoli, & Tavakoli-Hoseini, 2011; Emrani, Davoodnia, & Tavakoli-Hoseini, 2011; Khoshi, Davoodnia, & Prasada Rao Lingam, 2015; Moghadass, Davoodnia, Heravi, & Tavakoli-Hoseini, 2012; Nakhaei & Davoodnia, 2014; Taghavi-Khorasani & Davoodnia, 2015), herein, we report the results of our investigation on the application of IL₁ and IL₂ as catalysts in the synthesis of amidoalkyl naphthols through the one-pot, three-component reaction of β-naphthol, aryl aldehydes, and acetamide under neat conditions (Scheme 1).
2. Results and discussion

As a preliminary, we directed our studies toward examination of the effect of various parameters like catalyst composition, effect of solvent, and influence of temperature on the reaction of β-naphthol (1) (1.0 mmol), 4-chlorobenzaldehyde (2d) (1.0 mmol), and acetamide (3) (1.0 mmol) for the synthesis of compound 4d as the model reaction in the absence or presence of IL1 and IL2 as catalysts. A summary of the optimization experiments is provided in Table 1. First, to illustrate the need for catalyst in the reaction, the model reaction was studied in the absence of catalyst under solvent-free condition. The yield of the product was trace at 90°C after 60 min (Table 1, entry 1). Next, the reaction was performed in the presence of IL1 or IL2 in different solvents as well as under solvent-free conditions. Among the solvents tested, those being EtOH, MeOH, CH2Cl2, and MeCN, the reaction proceeded most readily to give the highest yield of the product 4d under solvent-free conditions. It was observed that the yield of the final product 4d increased with increasing amount of catalyst in the reaction mixture. The best result was obtained with 10 mol% of the catalyst under solvent-free conditions, which gave the desired product in 95 and 98% yields after 3 and 2 min at 90°C, respectively, for IL1 and IL2 (Table 1, entry 12). Further increase in temperature and IL1 or IL2 amount were found to have an inhibitory effect on formation of the product (Table 1, entries 13, 16, 17).

With the optimized conditions in hand, β-naphthol was reacted with acetamide and a wide variety of aromatic aldehydes using IL1 or IL2 (Table 2). As it can be seen, the reaction is effective with a variety of aromatic aldehydes with electron-donating or withdrawing substituents. Although the kind of aromatic aldehyde had no significant effect on the reaction, in most cases, but not all, aromatic aldehydes substituted with electron-withdrawing group or none reacted slightly faster than those with electron-donating groups and gave the higher yields of the products. Furthermore, both catalysts were highly efficient, and gave the desired amidoalkyl naphthols in high yields and short reaction times. However, as depicted, IL2 proved to be the better catalyst than IL1 in terms of yield and reaction time.

We also investigated recycling of the catalysts under solvent-free conditions using the model reaction. After completion of the reaction, the reaction mixture was cooled to room temperature, and warm distilled water was added. The product was collected by filtration, and washed repeatedly with warm distilled water. The combined filtrate was evaporated to dryness under reduced pressure. The residual ionic liquid was repeatedly washed with diethyl ether, dried under vacuum at 60°C, and used for the subsequent catalytic runs. The recovered catalyst worked well for up to five catalytic runs without any significant loss of its activity (95/98, 95/96, 93/95, 92/93, and 91/93% yields for IL1/IL2 catalysts in first to fifth use, respectively).
Table 1. Screening of reaction condition for synthesis of compound 4d catalyzed by IL<sub>1</sub> or IL<sub>2</sub>*

| Entry | Catalyst (mol%) | Solvent | T (°C) | Time (min) IL<sub>1</sub>/IL<sub>2</sub> | Isolated yield (%) IL<sub>1</sub>/IL<sub>2</sub> |
|-------|----------------|---------|--------|--------------------------------------|---------------------------------------------|
| 1     | –              | –       | 90     | 60/60 Trace/Trace                     | Trace/Trace                                 |
| 2     | 5              | –       | 70     | 6/5                                  | 60/68                                       |
| 3     | 5              | –       | 80     | 6/4                                  | 66/73                                       |
| 4     | 5              | –       | 90     | 5/3                                  | 73/76                                       |
| 5     | 5              | –       | 110    | 5/3                                  | 70/72                                       |
| 6     | 7              | –       | 70     | 5/5                                  | 67/72                                       |
| 7     | 7              | –       | 80     | 5/4                                  | 77/80                                       |
| 8     | 7              | –       | 90     | 4/3                                  | 85/89                                       |
| 9     | 7              | –       | 110    | 4/2                                  | 82/85                                       |
| 10    | 10             | –       | 70     | 5/4                                  | 75/79                                       |
| 11    | 10             | –       | 80     | 4/3                                  | 86/89                                       |
| 12    | 10             | –       | 90     | 3/2                                  | 95/98                                       |
| 13    | 10             | –       | 110    | 4/3                                  | 90/93                                       |
| 14    | 15             | –       | 70     | 6/5                                  | 70/74                                       |
| 15    | 15             | –       | 80     | 6/4                                  | 78/85                                       |
| 16    | 15             | –       | 90     | 5/3                                  | 88/92                                       |
| 17    | 15             | –       | 110    | 6/3                                  | 85/88                                       |
| 18    | 10             | EtOH    | Reflux | 35/25                                | 53/70                                       |
| 19    | 10             | MeOH    | Reflux | 45/35                                | 57/72                                       |
| 20    | 10             | CH<sub>2</sub>Cl<sub>2</sub> | Reflux | 40/30                                | 54/60                                       |
| 21    | 10             | MeCN    | Reflux | 30/20                                | 65/72                                       |

*Reaction conditions: β-naphthol (1) (1.0 mmol), 4-chlorobenzaldehyde (2d) (1.0 mmol), and acetamide (3) (1.0 mmol).

Table 2. IL<sub>1</sub> or IL<sub>2</sub> catalyzed synthesis of amidoalkyl naphthols (4a–k)*

| Entry | Ar         | Product | Time (min) IL<sub>1</sub>/IL<sub>2</sub> | Isolated yield (%) IL<sub>1</sub>/IL<sub>2</sub> | m.p. (°C) | lit. m.p. (°C) |
|-------|------------|---------|--------------------------------------|---------------------------------------------|----------|----------------|
| 1     | C<sub>6</sub>H<sub>5</sub> | 4a      | 4/2                                  | 94/97                                        | 242–244  | 240–242 (Kiasat et al., 2016) |
| 2     | 4-O<sub>2</sub>N<sub>4</sub>H<sub>2</sub> | 4b      | 4/2                                  | 89/91                                        | 246–248  | 242–244 (Kiasat et al., 2016) |
| 3     | 3-O<sub>2</sub>N<sub>4</sub>H<sub>2</sub> | 4c      | 6/3                                  | 86/90                                        | 239–241  | 241–243 (Kiasat et al., 2016) |
| 4     | 4-ClC<sub>6</sub>H<sub>5</sub> | 4d      | 3/2                                  | 95/98                                        | 228–230  | 225–227 (Kiasat et al., 2016) |
| 5     | 2-ClC<sub>6</sub>H<sub>5</sub> | 4e      | 4/3                                  | 86/91                                        | 206–208  | 204–205 (Shahriza et al., 2012) |
| 6     | 3-BrC<sub>6</sub>H<sub>5</sub> | 4f      | 4/2                                  | 90/91                                        | 227–229  | 229–230 (Shahriza et al., 2012) |
| 7     | 4-BrC<sub>6</sub>H<sub>5</sub> | 4g      | 5/2                                  | 92/94                                        | 227–228  | 230–232 (Davoodnia et al., 2014) |
| 8     | 4-FC<sub>6</sub>H<sub>5</sub> | 4h      | 4/3                                  | 93/95                                        | 224–226  | 226–228 (Davoodnia et al., 2014) |
| 9     | 4-MeC<sub>6</sub>H<sub>5</sub> | 4i      | 6/4                                  | 85/87                                        | 218–220  | 217–220 (Wang & Liang, 2011) |
| 10    | 4-MeO<sub>2</sub>C<sub>6</sub>H<sub>4</sub> | 4j      | 5/3                                  | 80/83                                        | 182–184  | 180–182 (Wang & Liang, 2011) |
| 11    | 3-Pyridyl   | 4k      | 6/5                                  | 86/89                                        | 190–192  | 192–194 (Bamoniri et al., 2014) |

*Reaction conditions: β-naphthol (1) (1.0 mmol), an aromatic aldehyde (2a-k) (1.0 mmol), acetamide (3) (1.0 mmol), IL<sub>1</sub> or IL<sub>2</sub> (0.1 mmol, 10 mol%), 90°C, solvent-free.
In accordance with the literature (Kiasat et al., 2016; Shahriasa et al., 2012), the suggested mechanism is described in Scheme 2. We believe that these ILs can act as Brønsted acids and therefore promote the reactions by increasing the electrophilic character of the electrophiles in the reaction. At first, ortho-quinone methide (o-QM) intermediate [II] is readily formed in situ by Knoevenagel condensation of \( \beta \)-naphthol (1) and aromatic aldehydes (2a-k) via the intermediate [I]. Subsequent Michael addition of acetamide (3) to the o-QM intermediate [II] afforded the final products 4a-k.

3. Conclusion
In conclusion, we showed that two newly synthesized Brønsted-acidic ILs, IL\(_1\) and IL\(_2\), efficiently catalyze the synthesis of amidoalkyl naphthols by increasing the electrophilic character of the electrophiles in the reaction \( \beta \)-naphthol, aryl aldehydes, and acetamide under solvent-free reactions. The kind of aldehyde had no significant effect on the reaction rates and products' yields. However, in general, electron-poor aldehydes reacted slightly faster than electron-rich ones and gave the higher yields of the products. Also, IL\(_2\) proved to be the better catalyst than IL\(_1\) in terms of yield and reaction time. Some advantages of this procedure are high yields, short reaction times, easy work-up, absence of volatile and hazardous solvents, and reusability of catalysts for a number of times without appreciable loss of activity.

4. Experimental
The IL\(_1\) and IL\(_2\) were synthesized according to our previous report (Dehghan et al., 2016). All chemicals were available commercially and used without additional purification. Melting points were recorded on a Stuart SMP3 melting point apparatus. The \(^1\)H NMR spectra were recorded with a Bruker 300 FT spectrometer.

4.1. General procedure for the synthesis of amidoalkyl naphthols (4a-k) catalyzed by IL\(_1\) or IL\(_2\)
A mixture of \( \beta \)-naphthol (1) (1.0 mmol), an aromatic aldehyde (2a-k) (1.0 mmol), acetamide (3) (1.0 mmol), and IL\(_1\) or IL\(_2\) (0.1 mmol, 10 mol \%) was heated in an oil bath at 90°C for 2–6 min. After completion of the reaction, monitored by TLC, the mixture was cooled to room temperature and warm distilled water was added. This resulted in the precipitation of the product, which was collected by filtration. The crude product was washed repeatedly with warm distilled water and then cold ethanol, and subsequently recrystallized from ethanol to give the pure products 4a-k in high yields. The products were characterized according to comparison of their melting points with those of authentic samples and for some of them by their \(^1\)H NMR spectral data.

4.2. Selected \(^1\)H NMR data
N-\((2\text{-hydroxynaphthalen-1-yl})(\text{phenyl})\text{methyl})\text{acetamide (4a):} \(^1\)H NMR (300 MHz, DMSO-\(d_6\)):
\[\delta\] 2.01 (s, 3H, CH\(_3\)), 7.10–7.45 (m, 9H, arom-H and CH\(_{sp^3}\)), 7.76–7.87 (m, 3H, arom-H and NH), 8.52 (d, 1H, \(J = 8.1\) Hz, arom-H), 10.08 (s br, 1H, OH).

N-\((2\text{-hydroxynaphthalen-1-yl})(4\text{-nitrophenyl})\text{methyl})\text{acetamide (4b):} \(^1\)H NMR (300 MHz, DMSO-\(d_6\)):
\[\delta\] 2.04 (s, 3H, CH\(_3\)), 7.18–7.33 (m, 3H, arom-H and CH\(_{sp^3}\)), 7.38–7.46 (m, 3H, arom-H), 7.78–7.88 (m, 3H, arom-H and NH), 8.16 (d, 2H, \(J = 9.0\) Hz, arom-H), 8.62 (d, 1H, \(J = 7.8\) Hz, arom-H), 10.01 (br, 1H, OH).
N-(4-Chlorophenyl)(2-hydroxynaphthalen-1-yl)methylacetamide (4d): ¹H NMR (300 MHz, DMSO-d₆): δ 2.03 (s, 3H, CH₃), 7.13–7.44 (m, 8H, arom-H and CH₆), 7.82 (t, 2H, J = 8.7 Hz, arom-H), 7.87 (br, 1H, NH), 8.54 (d, 1H, J = 8.1 Hz, arom-H), 10.11 (s, 1H, OH).

N-(3-Bromophenyl)(2-hydroxynaphthalen-1-yl)methylacetamide (4f): ¹H NMR (300 MHz, DMSO-d₆): δ 2.00 (s, 3H, CH₃), 7.12 (d, 2H, J = 6.9 Hz, arom-H), 7.18–7.45 (m, 6H, arom-H and CH₆), 7.77–7.90 (m, 3H, arom-H and NH), 8.51 (d, 1H, J = 8.4 Hz, arom-H), 9.69 (br, 1H, OH).

N-(4-Bromophenyl)(2-hydroxynaphthalen-1-yl)methylacetamide (4g): ¹H NMR (300 MHz, DMSO-d₆): δ 2.01 (s, 3H, CH₃), 7.08–7.32 (m, 5H, arom-H and CH₆), 7.40 (t, 1H, J = 8.1 Hz, arom-H), 7.46 (d, 2H, J = 8.4 Hz, arom-H), 7.76–7.88 (m, 3H, arom-H and NH), 8.51 (d, 1H, J = 8.1 Hz, arom-H), 10.04 (s, br, 1H, OH).

N-(2-hydroxynaphthalen-1-yl)(4-methoxyphenyl)methylacetamide (4j): ¹H NMR (300 MHz, DMSO-d₆): δ 2.00 (s, 3H, CH₃), 3.69 (s, 3H, OCH₃), 6.83 (d, 2H, J = 8.7 Hz, arom-H), 7.08–7.31 (m, 5H, arom-H and CH₆), 7.38 (t, 1H, J = 7.2 Hz, arom-H), 7.75–7.94 (m, 3H, arom-H and NH), 8.49 (d, 1H, J = 8.4 Hz, arom-H), 10.05 (br, 1H, OH).

N-(2-hydroxynaphthalen-1-yl)(pyridin-3-yl)methylacetamide (4k): ¹H NMR (300 MHz, DMSO-d₆): δ 2.03 (s, 3H, CH₃), 7.15–7.35 (m, 4H, arom-H and CH₆), 7.42 (t, 1H, J = 7.5 Hz, arom-H), 7.55 (d, 1H, J = 8.1 Hz, arom-H), 7.78–7.95 (m, 3H, arom-H and NH), 8.37–8.44 (m, 2H, arom-H), 8.57 (d, 1H, J = 8.1 Hz, arom-H), 10.14 (s, br, 1H, OH).

Supplemental data
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