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Silica-supported ionic liquid as highly efficient catalyst for one-pot synthesis of acenaphtho[1,2-*b*]furan compounds

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Abstract. Some new derivatives of acenaphtho[1,2-*b*]furan have been synthesized efficiently by one-pot reaction of (acenaphthylen-1-yloxy)trimethylsilane, various aldehydes and isocyanides at the presence of silica-supported ionic liquid.

Keywords. One-pot synthesis; acenaphtho[1,2-*b*]furan; silica-supported ionic liquid.

1. Introduction

During the past years, the synthesis of pharmacologically active furans, were widely known in nature,[1,2] have received much interest, probably, with the aim of preparing more biologically active derivatives of furans.[3–7] Isocyanide-based multi component reactions emerged in 1990s by Passerini,[8] are among the most important methods that have attracted more interest.[9–14]

As against traditional solvents, ionic liquids benefit from advantages such as undetectable vapour pressure, ease of being recycled, and their ability to dissolve many organic and inorganic substances and play specific tasks such as Brønsted acidic task-specific ionic liquids (BAILs).[15] In this regard, ionic liquids possessing HSO_4^- as counteranion, especially those have been immobilized on the solid surfaces, exhibit promising results in the area of catalysing reactions.[16–20]

In the previous studies,[21,22] we reported catalyst-free synthesis of acenaphtho [1,2-*b*]furan compounds employing isocyanide, aldehyde and silyl enol of acenaphthylen-1(2*H*)-one. However, chemical yields were not sustainable. Accordingly, in this study, we examined various ionic liquids to improve efficiency of our synthetic method. Among ionic liquids used, supported-ionic liquid (**d**) was found as the best choice and used for synthesis of compounds (**7a–k**) (scheme 1). In each case, efficiencies of catalysed and uncatalysed reaction were compared (table 1).

2. Experimental

2.1 *Materials and methods*

Compounds (**7c–g**) are new and characterized by IR, ^{1}HNMR, ^{13}CNMR and elemental micro analysing. ^{1}HNMR spectra were recorded on a Bruker AQSAVANCE-400 MHz spectrometer using TMS as an internal standard ($CDCl_3$ solution). ^{13}CNMR spectra were recorded on a Bruker AQSAVANCE-125 MHz spectrometer ($CDCl_3$ solution). IR spectra were recorded from KBr disk on the FT-IR Bruker Tensor. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer. (acenaphthylen-1-yloxy)trimethylsilane (**1**), supported and unsupported ionic liquids were prepared according to the literatures.[23,24]

2.2 *General procedure for preparation of acenaphtho[1,2-b]furan derivatives (**7a–k**)*

To a magnetically stirred mixture of aldehyde (1 mmol) and catalyst (0.01 mmol) in DMF (40 mL) at 0°C was drop-wise added to a solution of (acenaphthylen-1-yloxy)trimethylsilane (**1**) (1 mmol) in DMF (10 mL) over a 30 min period. Then, the mixture was allowed to warm to room temperature and treated with a solution of isocyanide (1.0 mmol) in DMF (10 mL). The mixture was refluxed for 10 h, cooled to room temperature, and filtered. The filtrate was extracted with dichloromethane

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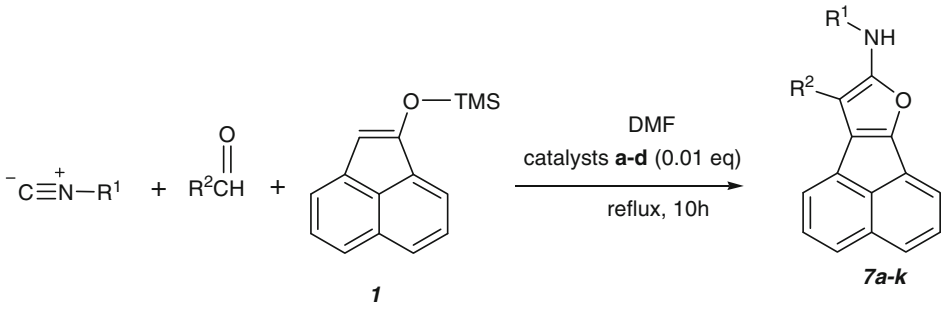
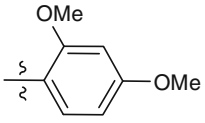
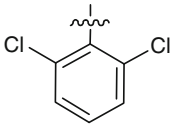
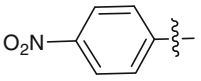
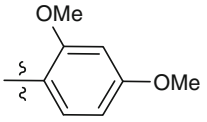
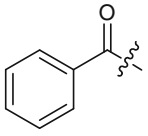
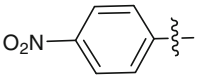
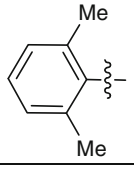
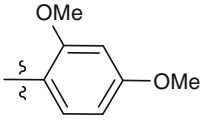
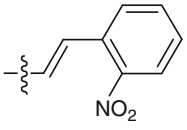
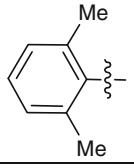
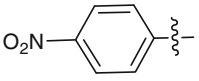
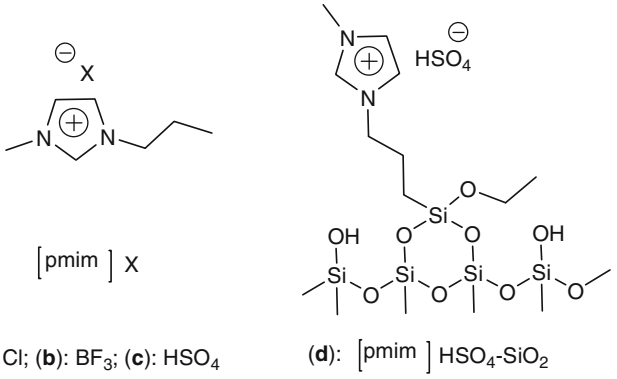


Entry (No)	R_1	R_2	Entry (No)	R_1	R_2
1 (7a)	c-hexyl		*7 (7g)*	c-hexyl	
2 (7b)	c-hexyl		*8 (7h)*	t-Butyl	
3 (7c)	c-hexyl		*9 (7i)*	t-Butyl	
4 (7d)	c-hexyl	H	*10 (7j)*		
5 (7e)	c-hexyl		*11 (7k)*		
6 (7f)	c-hexyl	n-Propyl			



Scheme 1. Preparation of compounds (**7a–k**) at the presence of ionic liquids (**a–d**).

Table 1. Comparison of ionic liquid-catalysed and uncatalysed routs for preparation of compounds (**7a–k**).

Entry	Compound	Time (h)/Yield (%)[a], catalysed by catalyst (d)	Time (h)/Yield (%)[a] uncatalysed
1	**7a**	10/97	24/89[22]
2	**7b**	10/95	24/93[22]
3	**7c**	10/94	24/76
4	**7d**	10/92	24/81
5	**7e**	10/91	24/75
6	**7f**	10/95	24/82
7	**7g**	10/92	24/80
8	**7h**	10/95	24/74[22]
9	**7i**	10/93	24/78[22]
10	**7j**	10/96	24/82[21]
11	**7k**	10/94	24/86[21]

[a]Isolated yields

(4 × 15 mL). After evaporation of solvent, the residue was washed with ether and crystallized from acetonitrile to give analytically pure product (**7a–k**).

2.2a *N-cyclohexyl-9-(2,4-dimethoxyphenyl)acenaphtho[1,2-b]furan-8-amine (**7a**)*: Brown powder (97%); m.p. 195–200°C; FT-IR (KBr) (ν_{max}, cm^{-1}): 3355 (N–H), 1230 (C–O), 1200 (C–O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 1.21–1.97 (10H, m, $5CH_2$), 3.52 (1H, m, N–CH), 3.75 (3H, s, OCH_3), 3.81 (3H, s, $-OCH_3$), 6.43 (1H, bs, NH), 6.62–6.72 (2H-m, Ar–H), 7.23 (1H, d, Ar–H), 7.63–7.81 (6H, m, Ar–H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 22.6, 28.7, 31.5, 49.6, 55.9, 56.2, 100.9, 107.1, 109, 117.8, 125.5, 127.3, 127.6, 128, 128.3, 129, 129.5, 133.5, 137.7, 139, 147.2, 158.4, 161.7; Anal. Calcd. for $C_{28}H_{27}NO_3$: C, 79.03; H, 6.40; N, 3.29%. Found: C, 78.84; H, 6.35; N, 3.23%.

2.2b *N-cyclohexyl-9-(4-nitrophenyl)acenaphtho[1,2-b]furan-8-amine (**7b**)*: Brown powder (95%); m.p. 209–214°C; FT-IR (KBr) (ν_{max}, cm^{-1}): 3281 (N–H), 1541 (N–O), 1360 (N–O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 1.24–2.09 (10H, m, $5CH_2$), 3.60 (1H, m, N–CH), 4.92 (1H, bs, NH), 7.65–7.86 (8H, m, Ar–H), 8.35 (2H, dd, Ar–H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 24.5, 26.1, 30.5, 30.8, 33.2, 51.3, 110.7, 121.4, 125.1, 125.7, 127.6, 127.9, 128.7, 129.1, 129.6, 130.3, 132.7, 135.1, 135.9, 137.4, 139.3, 141.5, 149.3, 154.8, 159.7, 175.4; Anal. Calcd. for $C_{26}H_{22}N_2O_3$: C, 76.08; H, 5.40; N, 6.82%. Found: C, 75.91; H, 5.34; N, 6.76%.

2.2c *(8-(Cyclohexylamino)acenaphtho[1,2-b]furan-9-yl)(phenyl)methanone (**7c**)*: Brown powder (94%); m.p. 181–184°C; FT-IR (KBr) (ν_{max}, cm^{-1}): 3350 (N–H), 1690 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 1.20–2.01 (10H, m, $5CH_2$), 3.53 (1H, m, N–CH), 6.16 (1H, bs, NH), 7.70–7.85 (5H, m, Ar–H), 7.90–8.04 (6H, m, Ar–H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 20.6, 26.6, 33.9, 47.1, 109.7, 120.2, 124.3, 125.7, 127.2, 127.9, 128.1, 128.5, 129.5, 133.1, 133.7, 135.1, 139.2, 149.3, 151.8, 190.7; Anal. Calcd. for $C_{27}H_{23}NO_2$: C, 82.42; H, 5.89; N, 3.56%. Found: C, 82.30; H, 5.81; N, 3.52%.

2.2d *N-cyclohexylacenaphtho[1,2-b]furan-8-amine (**7d**)*: Brown powder (92%); m.p. 161–163°C; FT-IR (KBr) (ν_{max}, cm^{-1}): 3364 (N–H); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 1.22–2.05 (10H, m, $5CH_2$), 3.51 (1H, m, N–CH), 4.76 (1H, s, =CH–), 6.09 (1H, bs, NH), 7.78 (2H, dd, Ar–H), 7.85–7.93 (4H, m, Ar–H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 24.3, 29.0, 33.6, 49.3, 104.8, 113.4, 127.3, 129.5, 129.9, 130.4, 131.7, 135.1, 138.9, 145.3, 150.2; Anal. Calcd. for $C_{20}H_{19}NO$: C, 83.01; H, 6.62; N, 4.84%. Found: C, 82.95; H, 6.60; N, 4.81%.

2.2e *9-(2-Nitrostyryl)-N-cyclohexylacenaphtho[1,2-b]furan-8-amine (**7e**)*: Brown powder (91%); m.p. 189–192°C; FT-IR (KBr) (ν_{max}, cm^{-1}): 3363 (N–H), 1535 (N–O), 1354 (N–O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H 1.29–2.01 (10H, m, $5CH_2$), 3.53 (1H, m, N–CH), 6.05 (1H, bs, NH), 7.16 (1H, d, J=12.4 Hz, ArC=H), 7.65 (1H, d, J=12.4 Hz, ArC=CH), 7.76–7.80 (5H, m, Ar–H), 8.01–8.12 (4H, m, Ar–H), 8.40 (1H, dd, Ar–H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 26.2, 31.6, 35.1, 53.3, 111.6, 124.6, 128.6, 129.1, 130.9, 131.6, 131.8, 132.2, 132.5, 133.1, 134.2, 137.6, 137.7, 139.3, 141.9, 143.2, 150.3, 151.7; Anal. Calcd. for $C_{28}H_{24}N_2O_3$: C, 77.04; H, 5.54; N, 6.42%. Found: C, 76.76; H, 5.47; N, 6.35%.

2.2f *N-cyclohexyl-9-propylacenaphtho[1,2-b]furan-8-amine (**7f**)*: Brown powder (95%); m.p. 174–176°C;

FT-IR (KBr) (ν_{max}, cm^{-1}): 3360 (N–H); ^1H NMR (400 MHz, $CDCl_3$) δ_H 0.92 (3H, t, J=7.8 Hz, CH_3–C–C), 1.25–2.11 (10H, m, $5CH_2$), 1.85 (2H, m, C–CH_2–C), 2.70 (2H, t, J=7.8 Hz, C–CH_2–C), 3.48 (1H, m, N–CH), 5.06 (1H, bs, NH), 7.69 (2H, dd, Ar–H), 7.82–7.92 (4H, m, Ar–H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 17.1, 20.3, 26.7, 28.6, 30.1, 33.6, 51.7, 113.1, 121.1, 127.6, 129.9, 131.7, 132.4, 132.4, 132.7, 137.3, 141.5, 141.8, 149.7; Anal. Calcd. for $C_{23}H_{25}NO$: C, 83.34; H, 7.60; N, 4.23%. Found: C, 83.21; H, 7.54; N, 4.12%.

2.2g *9-(2,6-Dichlorophenyl)-N-cyclohexylacenaphtho[1,2-b]furan-8-amine (**7g**)*: Brown powder (92%); m.p. 182–186°C; FT-IR (KBr) (ν_{max}, cm^{-1}): 3359 (N–H); ^1H NMR (400 MHz, $CDCl_3$) δ_H 1.22–2.00 (10H, m, $5CH_2$), 3.52 (1H, m, N–CH), 6.22 (1H, bs, NH), 7.23–7.32 (3H, m, Ar–H), 7.60 (2H, m, Ar–H), 7.77–7.89 (4H, m, Ar–H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 25.5, 31.3, 34.4, 52.5, 113.9, 129.6, 131.4, 131.6, 131.7, 132.4, 132.4, 133.1, 134.8, 136.7, 136.9, 138.7, 140.9, 142.5; Anal. Calcd. for $C_{26}H_{21}Cl_2NO$: C, 71.89; H, 4.87; N, 3.22%. Found: C, 71.74; H, 4.81; N, 3.17%.

2.2h *N-tert-butyl-9-(2,4-dimethoxyphenyl)acenaphtho[1,2-b]furan-8-amine (**7h**)*: Brown powder (95%); m.p. 135–138°C; FT-IR (KBr) (ν_{max}, cm^{-1}): 3364 (N–H), 1210 (C–O), 1200 (C–O); ^1H NMR (400 MHz, $CDCl_3$) δ_H 1.18 (9H, s, $C(CH_3)_3$), 3.70 (3H, s, OCH_3), 3.74 (3H, s, –OCH_3), 5.41 (1H, bs, NH), 6.59–6.69 (2H–m, Ar–H), 7.19 (1H, d, Ar–H), 7.59–7.71 (6H, m, Ar–H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 30.7, 51.3, 57.6, 57.9, 102.6, 108.8, 110.7, 119.5, 127.2, 129, 129.3, 129.7, 130.0, 130.7, 131.2, 135.2, 139.4, 140.7, 148.9, 160.1, 163.4; Anal. Calcd. for $C_{26}H_{25}NO_3$: C, 78.17; H, 6.31; N, 3.51%. Found: C, 78.01; H, 6.24; N, 3.45%.

2.2i *N-tert-butyl-9-(4-nitrophenyl)acenaphtho[1,2-b]furan-8-amine (**7i**)*: Brown powder (93%); m.p. 146–148°C; FT-IR (KBr) (ν_{max}, cm^{-1}): 3364 (N–H), 1550 (N–O), 1343 (N–O); ^1H NMR (400 MHz, $CDCl_3$) δ_H 1.17 (9H, s, $C(CH_3)_3$), 6.43 (1H, bs, NH), 7.75–7.80 (8H, m, Ar–H), 8.29 (2H, dd, Ar–H); 7.76–8.30 (10H, m, Ar–H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 31.8, 53.9, 113.1, 123.7, 127.5, 128.1, 130.0, 130.3, 131.5, 132, 132.7, 135.1, 137.5, 138.3, 139.8, 141.7, 143.9, 151.7, 157.2, 162.1, 177.8; Anal. Calcd. for $C_{24}H_{20}N_2O_3$: C, 74.98; H, 5.24; N, 7.29%. Found: C, 74.71; H, 5.16; N, 7.19%.

2.2j *9-(2,4-Dimethoxyphenyl)-N-(2,6-dimethylphenyl)acenaphtho[1,2-b]furan-8-amine (**7j**)*: Brown powder (96%); m.p. 163–167°C; FT-IR (KBr) (ν_{max}, cm^{-1}): 3451 (N–H), 1130 (C–O), 1200 (C–O); ^1H NMR (400 MHz, $CDCl_3$) δ_H 2.36 (6H, s, 2Me), 3.57 (3H, s, OCH_3), 3.62 (3H, s, -OCH_3), 6.74 (1H, bs, NH), 6.43–6.70 (5H, m, Ar–H), 7.26 (1H, dd, Ar–H), 7.60 (2H, m, Ar–H), 7.69–7.84 (4H, m, Ar–H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 18.4, 58.7, 59.5, 104.7, 110.9, 112.8, 121.5, 121.6, 128.4, 129.8, 130.2, 130.5, 130.9, 131.2, 131.8, 133.9, 134.4, 138.4, 142.6, 142.6, 144.8, 152.1, 163.3, 166.6; Anal. Calcd. for $C_{30}H_{25}NO_3$: C, 80.51; H, 5.63; N, 3.13%. Found: C, 80.46; H, 5.58; N, 3.02%.

2.2k *N-(2,6-Dimethylphenyl)-9-(4-nitrophenyl)acenaphtho[1,2-b]furan-8-amine (**7k**)*: Brown powder (94%); m.p. 142–144°C; FT-IR (KBr) (ν_{max}, cm^{-1}): 3237 (N–H), 1501 (N–O), 1418 (N–O); ^1H NMR (400 MHz, $CDCl_3$) δ_H 2.21 (6H, s, 2Me), 6.36 (1H, bs, NH), 6.58 (1H, dd, Ar–H) 6.72 (2H, m, Ar–H), 7.66 (2H, m, Ar–H), 7.74–7.90 (6H, m, Ar–H), 8.27 (2H, d, Ar–H); ^{13}C NMR (100 MHz, $CDCl_3$) δ_C 22.4, 113.1, 122.7, 125.7, 129.6, 129.7, 130.5, 130.4, 130.7, 130.8, 131.9, 134.0, 134.1, 138.6, 142.2, 142.8, 145.2, 147.6, 152.3, 153.5; Anal. Calcd. for $C_{28}H_{20}N_2O_3$: C, 77.76; H, 4.66; N, 6.48%. Found: C, 77.58; H, 4.43; N, 6.41%.

3. Results and discussions

Three ionic liquid catalysts (10% mol) of [pmim]Cl (1-propyl-3-methylimidazolium–Cl) (**a**), [pmim]BF_4 (1-propyl-3-methylimidazolium–BF_4) (**b**) and [pmim]HSO_4 (1-propyl-3-methylimidazolium–HSO_4) (**c**) (scheme 1), possessing different counteranions, were evaluated for catalysing the reaction of an equal stoichiometric ratio of silyl enol of acenaphthylen-1(2*H*)-one (**1**), 2,4-dimethoxybenzaldehyde and isocyanocyclohexane. As a result, catalyst (**c**) was found as the best choice (table 2).

In order to reveal the plausible effect of supporting on the catalytic behaviour of [pmim]HSO_4 (**c**), it was immobilized on the modified silica. The use of supported catalyst, [pmim]HSO_4–SiO_2 (**d**) (scheme 1), resulted in a significant improvement in reaction times and catalytic yields attributed plausibly to the participation of SiO_2 in the catalytic process (table 2, entry 2). However, this hypothesis was established to some extent when the silica support was used as catalyst under the same reaction conditions. As can be in table 2 (compare entries 1 and 6), efficiencies of the reactions

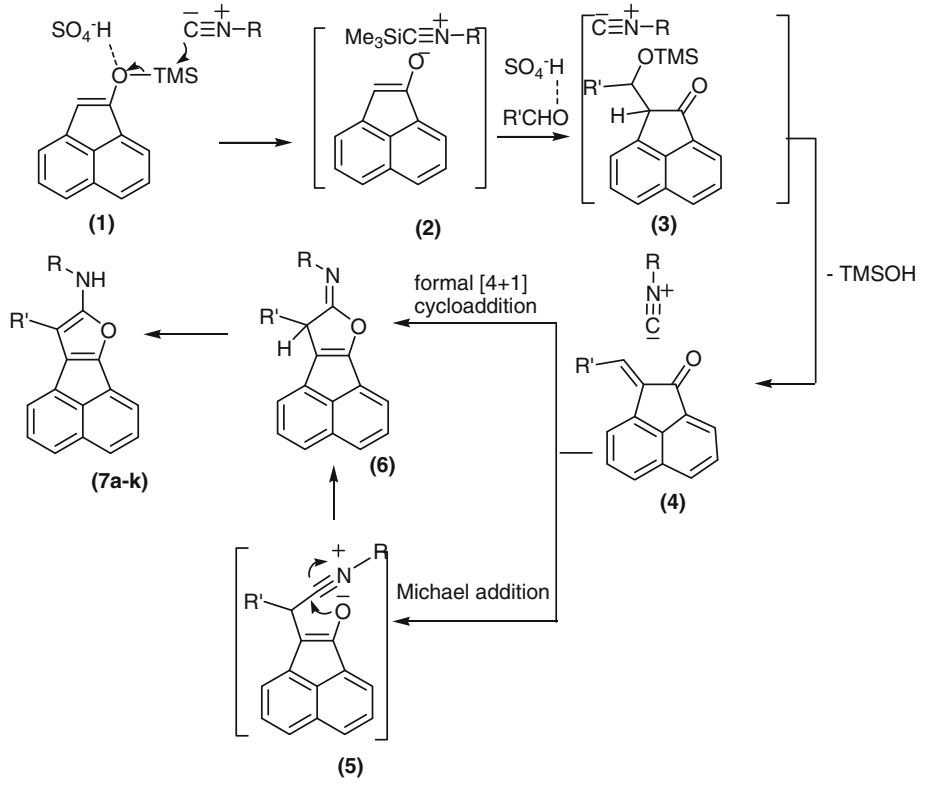
Table 2. Catalytic efficiencies of ionic liquids (**a–d**) for the preparation of compound (**7a**).[a]

Entry	Catalyst	Time (h)	Yield (%)[b]
1	------	24	89
2	[pmim]HSO_4-SiO_2	10	95
3	[pmim]HSO_4	14	92
4	[pmim]BF_4	18	90
5	[pmim]Cl	18	88
6	Silica support	20	87

[a]Reaction conditions: 1.0 equiv. of silyl enol of acenaphthylen-1(2*H*)-one, 1.0 equiv. of 2,4-dimethoxybenzaldehyde, 1.0 equiv. of isocyanocyclohexane, 10 mol% of catalyst, 60 mL of DMF as solvent and at refluxing condition.
[b]Isolated yields

in terms of times and yields compared with uncatalysed system were found better. So, supported ionic liquid (**d**) was used to assess the generality and scope of reaction with respect to the aldehyde and isocyanide components (table 1). As a result, the reactions proceeded well with various aromatic and aliphatic aldehydes, such as α, β-unsaturated aldehydes, and electronically sufficient and deficient aldehydes. Moreover, employing sterically hindered isocyanides also could not prevent the reactions to afford their good yields (table 1, entries 8–11). As can be seen, all chemical yields and reaction times were improved in the presence of ionic liquid (**d**) compared with those we obtained under uncatalysed conditions.

With the aim of investigating the effects of temperature and solvent, the reactions were carried out at the various temperatures and in the different solvents such as CH_2Cl_2, $CHCl_3$, ethanol, CH_3CN, and finally refluxed DMSO were found as the best reaction conditions.



Scheme 2. Proposed mechanism for the one-pot three-component synthesis of compounds **7a–k**.

As established in our previous studies,[21,22] at the first step, isocyanide attacks on O–$SiMe_3$ bond of silyl enol ether (**1**) to generate active enol ion pair complex (**2**). Then, α, β-unsaturated ketone (**4**) generated after condensation of enol (**2**) and aldehyde can produce iminolactone intermediate (**6**) either via a formal [4+1]-cycloaddition reaction or a Michael-type addition with the isocyanide. Finally, the intermediate (**6**) isomerizes to form aminofuran heteroaromatic moiety (**7**) (scheme 2).

4. Conclusion

A novel multicomponent approach for the synthesis of a series of new acenaphthofuran derivatives utilizing the supported ionic liquid catalyst has been elaborated. The efficient catalysing of used ionic liquid in the synthesis of acenaphtho[1,2-*b*]furans led to high chemical yields as well as short reaction times.

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