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Note

Ionic liquid promoted synthesis of β-enamino ketones and esters under microwave irradiation

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A facile amination of 1,3-dicarbonyl compounds with amines or ammonium salts has been achieved under microwave irradiation in the presence of acidic ionic liquid 1-butyl-3-methylimidazolium hydrogen sulphate. The method has the advantage of solvent free reactions, short reaction time and mild conditions.

Keywords: Solvent free, β-enaminone, β-enamino ester, ionic liquid, microwave

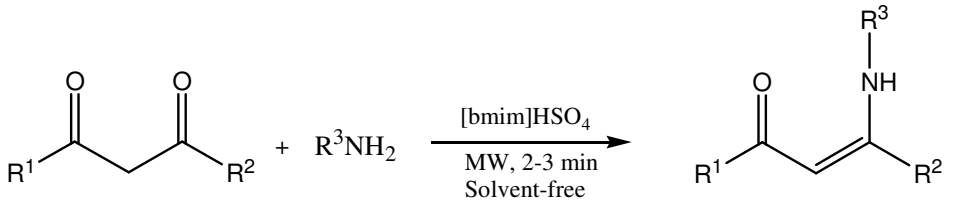
β-Enaminones and β-enamino esters are useful intermediates in organic synthesis as synthons for the construction of biologically active compounds such as dopamine auto-receptor agonists[1], acetylcholinestersase inhibitors[2], oxytocin antagonists[3] and anticonvulsants[4]. On the other hand, substituted β-enamino ketones and esters are useful intermediates for the synthesis of heterocycles like pyridinones[5], quinolones[6], oxazoles[7], pyrroles[8], isoxazole derivatives[9], tetrahydrobenzo-azines[10] and polyheterocycles[11]. The enamino ketone moiety has attracted much attention because of its use as an intermediate for the preparation of part of the side chain of taxol[12]. Several methods for the preparation of β-enamino ketones and esters have been reported. The common method used is the condensation between β-dicarbonyl compounds and amines in the presence of benzene by azeotropic removal of water[13]. Some successful modifications have been reported such as the use of catalysts like B_2O_3/Al_2O_3 (Ref 14), $ZrCl_4$ (Ref 15), $H_2SO_4.SiO_2$ (Ref 16), $[VO(acac)_2]$ (Ref 17), I_2 (Ref 18), $HClO_4.SiO_2$ (Ref 19), Gold(I)/Silver(I) (Ref 20), $Fe(HSO_4)_3.SiO_2$ (Ref 21), $Ni(OAc)_2$ (Ref 22), $P_2O_5.SiO_2$ (Ref 23), $Ga(OTf)_3$ (Ref 24), montmorillonite-K10-microwave (Ref 25), $Er(OTf)_3$ (Ref 26), SiO_2Cl and ionic liquid $[Hbim]BF_4$ (Ref 27). Most of these methods have drawbacks such as long reaction time, use of hazardous solvents and expensive and stoichiometric catalyst. To overcome all these drawbacks we have developed a new method using ionic liquid under microwave irradiation. The acidic ionic liquid 1-butyl-3-methylimidazolium hydrogen sulphate $[bmim]HSO_4$ has not been explored in organic reactions to a great extent even though it holds great potential due to its Brϕnsted acidity and highly polar nature that augments absorption of microwave energy. In continuation with our studies towards exploring the use of $[bmim]HSO_4$ (Ref 28) we synthesized of β-enaminones and esters by using $[bmim]HSO_4$ as catalyst under microwave irradiation.

Results and Discussion

Enamination of β-dicarbonyl compounds was carried out by mixing equimolar amounts of β-dicarbonyl compound, amine and catalytic amount of ionic liquid followed by exposure to microwave. To extend the scope of the reaction as a general and practical procedure for enamination, the reaction series of aryl and aliphatic amines were carried out with pentane-2,4-dione and ethyl 3-oxobutanoate under solvent free conditions at 70-75°C in the presence of $[bmim]HSO_4$ (0.7 mmol) (**Scheme I**). The reactions were completed in 2-3 min affording excellent yields of the enaminones and enamino esters (**Table I**).

The amination of 1,3-dicarbonyl compounds was carried out with different ammonium salts. In all the cases it was found that good to moderate yields were obtained except with ammonium chloride (**Scheme II**).

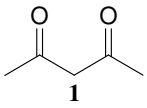
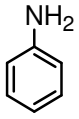
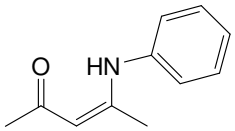
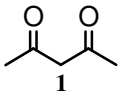
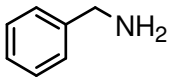
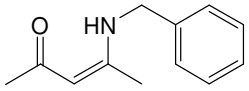
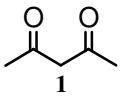
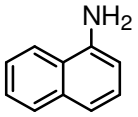
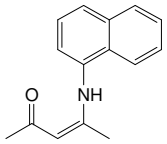
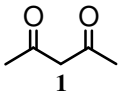
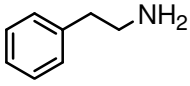
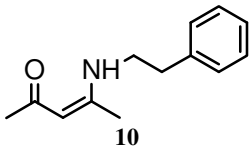
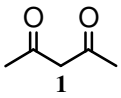
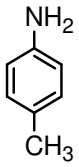
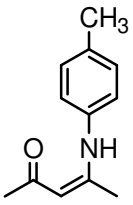
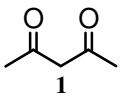
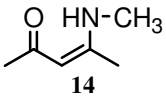
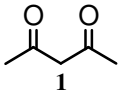
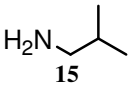
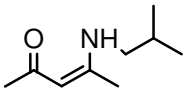
For the determination of the effective amount of $[bmim]HSO_4$, the reaction of pentane-2,4-dione with aniline was considered as the model. When the reactions were carried out with lower amounts of catalyst (0.1 and 0.4 mmol) either trace amounts of product were formed or incomplete conversion of the starting materials to the product was observed after 3 min at 75°C (**Table II**). Excellent conversion to 4-phenyl-amino-pent-3-en-2-one took place with 0.7 mmol and 1.0 mmol of $[bmim]HSO_4$ after 3 min at 75°C. No significant reduction in the time was observed in increasing the catalyst loading to 1.0 mmol. Thus



$R^1 = CH_3$, OEt, $R^2 = CH_3$ and R^3 = Ar, Alkyl

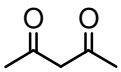
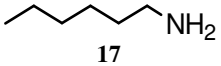
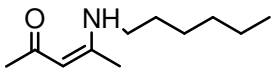
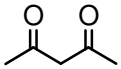
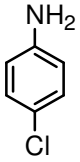
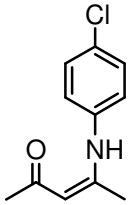
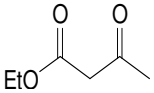
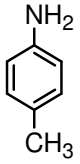
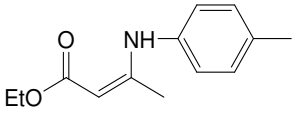
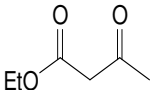
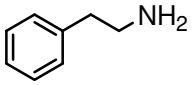
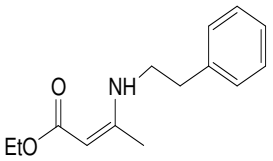
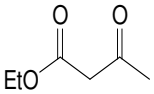
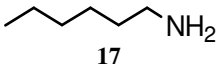
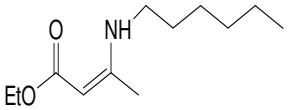
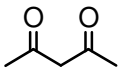
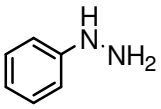
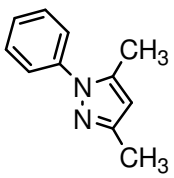
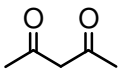
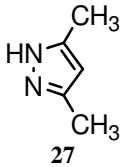
Scheme I

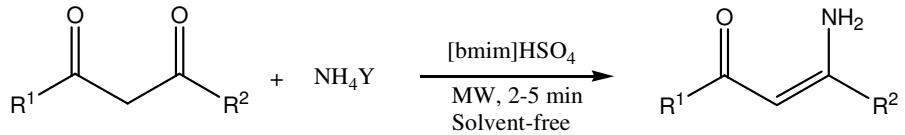
Table I — Condensation of 1,3-dicarbonyl compounds with amines

Reactant		Product	Time (min)	Yield (%)
 **1**	+  **3**	 **4**	3	94
 **1**	+  **5**	 **6**	3	90
 **1**	+  **7**	 **8**	3	95
 **1**	+  **9**	 **10**	3	96
 **1**	+  **11**	 **12**	2	98
 **1**	+ CH_3NH_2 **13**	 **14**	3	73
 **1**	+  **15**	 **16**	3	70

Contd —

Table I — Condensation of 1,3-dicarbonyl compounds with amines — *Contd*

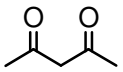
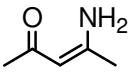
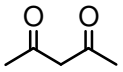
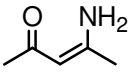
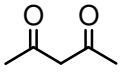
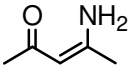
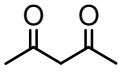
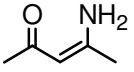
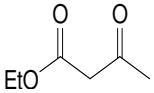
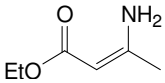
Reactant		Product	Time (min)	Yield (%)
 **1**	+  **17**	 **18**	3	90
 **1**	+  **19**	 **20**	3	84
 **2**	+  **11**	 **21**	2	95
 **2**	+  **9**	 **22**	2	92
 **2**	+  **17**	 **23**	2	85
 **1**	+  **24**	 **25**	2	92
 **1**	+ $NH_2NH_2.H_2O$ **26**	 **27**	2	95



$R^1 = CH_3$, OEt, $R^2 = CH_3$

Scheme II

Table II — Amination of 1,3-dicarbonyl compounds

Reactants			Product	Time (min)	Yield (%)
 **1**	+	$(NH_4)_2CO_3$ **28**	 **29a**	3	85
 **1**	+	$(COONH_4)_2.H_2O$ **30**	 **29b**	3	95
 **1**	+	CH_3COONH_4 **31**	 **29c**	3	96
 **1**	+	NH_4Cl **32**	 **29d**	5	10
 **2**	+	CH_3COONH_4 **31**	 **33**	2	80

In the column of product number (a, b, c, d) are the product number of same compound prepared by different reactant.

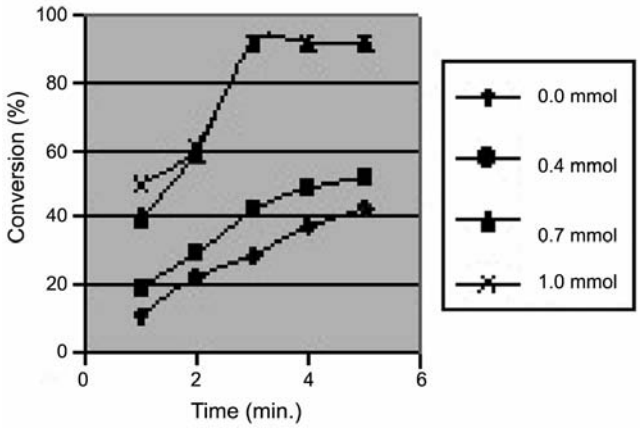


Figure 1 — Graphical representation of catalyst loading

effective catalyst loading was found to be 0.7 mmol (**Figure 1**).

To investigate, reusability of the catalyst, we have taken a model reaction of pentane 2,4-dione and aniline (**Table III**). After completion of the reaction, the residual ionic liquid was washed with diethyl ether for 20 min and ethereal layer was decanted. The catalyst was dried under reduced pressure. We were able to recycle the catalyst twice under microwave irradiation after that there was slight decrease in the yield of the product.

Table III — Reusability of catalyst

Run	Yield(%)
1	94
2	92
3	84

Experimental Section

Melting points were determined with Sunbeam melting point apparatus and are uncorrected. IR spectra were recorded on a Perkin Elmer model 1430 spectrometer. ^1H and ^{13}C NMR spectra were recorded using Joel 300 MHz spectrometer. Chemical shifts (δ) are reported in ppm with tetramethylsilane as internal standard. Microwave induced reactions were carried out in Monomode Plazmatronika RM 2001 PC.

General procedure for enamination of β-dicarbonyl compounds with amines

A mixture of 1,3-dicarbonyl compound (5.0 mmol), amine (5.0 mmol) and bmim[HSO_4] (0.7 mmol) in a 25 mL conical flask was irradiated at 70-75°C for 3 min under microwave. After completion of the reaction as indicated by TLC, the reaction mixture was extracted with DCM (2 × 10 mL) and washed with brine. The organic layer was dried over anhydrous Na_2SO_4. Removal of the solvent under

reduced pressure followed by column chromatography yielded pure product.

4-Phenylamino-pent-3-en-2-one, 4 (Ref 20, 24). m.p. 45-48°C. IR (neat): 1568, 1596, 3346 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.96 [s, 3H, C*H*$_3$-C(NH)=], 2.08 [s, 3H, C*H*$_3$-C(=O)-], 5.11 (s, 1H,-C*H*=), 7.04-7.16 (m, 3H, Ar*H*), 7.27-7.32 (m, 2H, Ar*H*), 12.45 (bs, 1H,-N*H*, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 19.6, 28.8, 97.4, 124.4, 125.2, 128.8, 138.6, 159.2, 195.2.

4-Benzylamino-pent-3-en-2-one, 6 (Ref 18, 20). IR (neat): 1515, 1606, 3308 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.88 [s, 3H, C*H*$_3$-C(NH)=], 1.99 [s, 3H, C*H*$_3$-C(=O)-], 4.43 (d, *J* = 6.3 Hz, 2H,-NH-C*H*$_2$-Ph), 4.98 (s, 1H,-C*H*=), 7.19-7.33 (m, 5H, Ar*H*), 11.14 (bs, 1H,-N*H*, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 18.5, 28.5, 46.3, 95.7, 126.2, 127.4, 128.4, 137.8, 162.1, 194.0.

4-(Naphthalene-1-ylamino)-pent-3-en-2-one, 8 (Ref 20). IR (neat): 1574, 1620, 3410 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.83 [s, 3H, C*H*$_3$-C(NH)=], 2.11 [s, 3H, C*H*$_3$-C(=O)-], 5.20 (s, 1H,-C*H*=), 7.17-7.20 (m, 1H, Ar*H*), 7.33-7.38 (m, 1H, Ar*H*), 7.42-7.50 (m, 2H, Ar*H*), 7.65-7.68 (m, 1H, Ar*H*), 7.76-7.79 (m, 1H, Ar*H*), 7.97-7.99 (m, 1H, Ar*H*), 12.73 (bs, 1H,-N*H*, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 19.3, 28.8, 97.3, 122.6, 122.9, 124.8, 126.0, 126.2, 126.5, 128.2, 130.0, 134.1, 134.8, 160.6, 195.4.

4-Phenethylamino-pent-3-en-2-one, 10 (Ref 29). IR (neat): 1574, 1608, 3421 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.73 [s, 3H, C*H*$_3$-C(NH)=], 1.93 [s, 3H, C*H*$_3$-C(=O)-], 2.82 (t, *J* = 7.2 Hz, 2H,-C*H*$_2$-Ph), 3.42 (q, *J* = 7.2 Hz, 2H,-NH-C*H*$_2$-CH$_2$-), 4.83 (s, 1H,-C*H*=), 7.13-7.27 (m, 5H, Ar*H*), 10.85 (bs, 1H,-N*H*, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 18.4, 28.5, 36.8, 44.5, 95.2, 126.4, 128.4, 128.6, 138.1, 161.8, 194.0.

4-*p*-Tolylamino-pent-3-en-2-one, 12 (Ref 20). m.p. 63-65°C. IR (neat): 1594, 1620, 3405 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.94 [s, 3H, C*H*$_3$-C(NH)=], 2.04 [s, 3H, C*H*$_3$-C(=O)-], 2.32 (s, 3H, C*H*$_3$-Ph), 5.08 (s, 1H,-C*H*=), 6.96 (d, *J* = 8.4 Hz, 2H, Ar*H*), 7.09 (d, *J* = 8.4 Hz, 2H, Ar*H*), 12.37 (bs, 1H,-N*H*, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 19.6, 21.0, 29.4, 97.5, 124.3, 129.2, 136.1, 136.7,160.1, 195.4.

4-Methylamino-pent-3-en-2-one, 14 (Ref 30). m.p. 45-47°C. IR (neat): 1576, 1610, 3418 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.90 [s, 3H, C*H*$_3$-C(NH)=], 1.91 [s, 3H, C*H*$_3$-C(=O)-], 2.92 (d, *J* = 5.1 Hz, 3H, C*H*$_3$-NH-), 4.90 (s, 1H,-C*H*=), 10.41 (bs, 1H,-N*H*, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 17.7, 27.8, 28.5, 94.4, 162.7, 193.1.

4-Isobutylamino-pent-3-en-2-one, 16. IR (neat): 1579, 1609, 3374 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.04 (d, *J* = 6.6Hz, 6H, 2 × C*H*$_3$), 1.80 [m, 1H, -C*H*-(CH$_3$)$_2$],1.95 [s, 3H, C*H*$_3$-C(NH)=], 2.21 [s, 3H, C*H*$_3$-C(=O)-], 3.04 (d, *J* = 6.6Hz, 2H,-NH-C*H*$_2$-CH<), 4.91 (s, 1H,-C*H*=), 10.96 (bs, 1H,-N*H*, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 18.3, 19.8, 28.3, 28.9, 50.3, 94.8, 162.4, 193.9.

4-Hexylamino-pent-3-en-2-one, 18 (Ref 26). IR (neat): 1574, 1609, 3395 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 0.89 (t, *J* = 6.6Hz, 3H, C*H*$_3$-), 1.26-1.60 (m, 8H, saturated methylene protons), 1.91 [s, 3H, C*H*$_3$-C(NH)=], 1.97 [s, 3H, C*H*$_3$-C(=O)-], 3.18 (t, *J* = 6.6Hz, 2H,-NH-C*H*$_2$-), 4.91 (s, 1H,-C*H*=), 10.83 (bs, 1H,-N*H*-, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 13.8, 18.4, 22.3, 26.3, 28.4, 29.9, 31.2, 42.7, 94.8, 162.5, 194.0.

4-(4-Chloro-phenylamino)-pent-3-en-2-one, 20 (Ref 20). m.p. 58-60°C. IR (neat): 1563, 1613, 3452 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.91 [s, 3H, C*H*$_3$-C(NH)=], 2.02 [s, 3H, C*H*$_3$-C(=O)-], 5.08 (s, 1H,-C*H*=), 6.91-7.33 (m, 4H, Ar*H*), 12.35 (bs, 1H, -N*H*, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 19.8, 29.2, 97.9, 125.7, 128.8, 136.7, 159.3, 196.8.

Ethyl 3-(*p*-tolylamino)but-2-enoate, 21 (Ref 20). IR (neat): 1607,1651, 3360 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.27 (t, *J* = 7.2 Hz, 3H, O-CH$_2$-C*H*$_3$), 1.94 (s, 3H, C*H*$_3$-C(NH)=), 2.32 (s, 3H, C*H*$_3$-Ph), 4.11 (q, *J* = 7.2 Hz, 2H, O-C*H*$_2$-), 4.60 (s, 1H,-C*H*=), 6.94 (d, *J* = 8.1 Hz, 2H, Ar*H*), 7.08 (d, *J* = 8.1 Hz, 2H, Ar*H*), 10.27 (bs, 1H,-N*H*, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.5, 20.0, 20.7, 58.3, 85.4, 124.5, 129.4, 134.4, 136.6, 158.8, 170.1.

Ethyl 3-(phenethylamino)but-2-enoate, 22 (Ref 19). IR (neat): 1608, 1648,3284 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.21 (t, *J* = 7.2 Hz, 3H, O-CH$_2$-C*H*$_3$), 1.75 (s, 3H, C*H*$_3$-C(NH)=), 2.80 (t, *J* = 7.2 Hz, 2H,-C*H*$_2$-Ph), 3.38 (q, *J* = 7.2 Hz, 2H, -NH-C*H*$_2$-CH$_2$-), 4.03 (q, *J* = 7.2 Hz, 2H, O-C*H*$_2$-), 4.34 (s, 1H,-C*H*=), 7.13-7.24 (m, 5H, Ar*H*), 8.61 (bs, 1H,-N*H*, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 14.6, 19.1, 37.3, 44.6, 58.0, 82.5, 126.4, 128.5, 128.6, 138.4, 161.0, 170.2.

Ethyl 3-(hexylamino)but-2-enoate, 23 (Ref 26). IR (neat): 1608, 1652, 3358 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 0.91 (t, *J* = 6.6 Hz, 3H, CH$_3$-), 1.19-1.60 (m, 11H, O-CH$_2$-C*H*$_3$, saturated methylene protons), 1.88 (s, 3H, C*H*$_3$-C(NH)=), 3.18 (t, J = 6.8 Hz, 2H,

-NH-CH_2-), 4.00 (q, J = 7.2 Hz, 2H, O-CH_2-), 4.30 (s, 1H,-CH=), 8.52 (bs, 1H,-NH-, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 13.9, 14.6, 19.2, 22.4, 26.4, 30.4, 31.4, 42.9, 57.9, 82.1, 161.1, 170.2.

3,5-Dimethyl-1-phenyl-1H-pyrazole, 25 (Ref 31). IR (neat): 1556, 1599 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 2.27 (s, 6H, 2× CH_3-), 5.93 [s, 1H, =CH-C(CH_3)=], 7.28-7.39 (m, 5H, ArH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 12.1, 13.2, 106.8, 124.4, 126.9, 128.6, 138.8, 139.6, 148.4.

3,5-Dimethyl-1H-pyrazole, 27 (Ref 32). m.p. 105-107°C IR (neat): 1505, 1605, 3195 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 2.27 (s, 6H, 2 × CH_3-), 5.74 [s, 1H, =CH-C(CH_3)=], 10.96 (bs, 1H,-NH-, D_2O exchangeable); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 12.3, 104.0, 143.8.

General procedure for enamination of β-dicarbonyl compounds with ammonium salts

4-Amino-pent-3-en-2-one, 29 (Ref 33). A mixture of pentane-2,4-dione (5.0 mmol), ammonium salts (5.0 mmol) and bmim[HSO_4] (0.7 mmol) in a 25 mL conical flask was irradiated at 70-75°C for 3 min under microwave. After completion of the reaction as indicated by TLC, the reaction mixture was extracted with dichloromethane (2 × 10 mL) and washed with brine. The organic layer was dried over anhydrous sodium sulphate. Removal of solvent under reduced pressure followed by column chromatography yielded pure product.

m.p. 33-34°C. IR (neat): 1596, 1693, 3357 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.84 [s, 3H, CH_3-C(NH)=], 1.92 [s, 3H, CH_3-C(=O)-], 4.90 (s, 1H, -CH=), 9.61 (bs, 2H,-NH_2); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.9, 29.0, 95.3, 161.2, 195.7.

Ethyl 3-aminobut-2-enoate, 33 (Ref 33). IR (neat): 1654, 1717, 3457 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.17 (t, J = 7.2 Hz, 3H, O-CH_2-CH_3), 2.16 (s, 3H, CH_3-C(NH)=), 4.05 (q, J = 7.2 Hz, 2H, O-CH_2-), 4.30 (s, 1H,-CH=), 7.78 (bs, 2H,-NH_2); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 4.3, 21.8, 58.0, 83.5, 159.9, 169.8.

Conclusion

In conclusion, a new method has been projected for the solvent free enamination of β-dicarbonyl compounds in excellent yields. Its notable features are solvent free conditions, short reactions times, simple experimental procedure and reusability of the catalyst.

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