$FeCl_3$-PTSA co-catalysed highly regio- and stereo-selective synthesis of β-functionalised enamine derivatives

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β-Enaminone derivatives are useful synthetic precursors. β-*N*-Substituted (*E*)-enaminones and β-N-substituted (*E*)-aminoacrylates were synthesised with high regio- and stereo-selectivity via using the co-catalytic system of $FeCl_3$/PTSA, which also provides a new way to the formation of C–O bond.

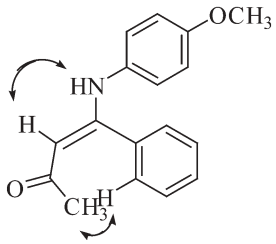
Keywords: high regio-selectivity; high stereo-selectivity, co-catalytic system, β-N-substituted (*E*)-enaminones, β-N-substituted (*E*)-aminoacrylates

β-Enaminone derivatives[1–10] are useful synthetic precursors[11,12] and their utilisation in organic synthesis is of great interest[13–22] due to the presence of the nucleophilic character of the enamine moiety and the electrophilic character of the enone moiety. A variety of biologically active compounds, such as heterocycles,[23–26] aminoacids,[27,28] alkaloids,[29–32] have been prepared from β-N-substituted enaminones and aminoacrylates taking advantage of their electronic properties. In this regard, there are many reports for the synthesis of β-functionalised enamines.[33–36] However, some of them usually present significant limitations, such as tedious workup, harsh nature of reaction conditions, low yields, long reaction times, or poor scope of substrates. In conjunction with our interest in exploring the synthetic potential of β-functionalised enamines, we now report an efficient and straightforward protocol under mild conditions to construct β-N-substituted (*E*)-enaminones and (*E*)-aminoacrylates (Scheme 1). The structure of the product **3ad** has been confirmed by ^{1}H NMR, ^{13}C NMR and the NMR-NOE further verified its configuration (Scheme 2).

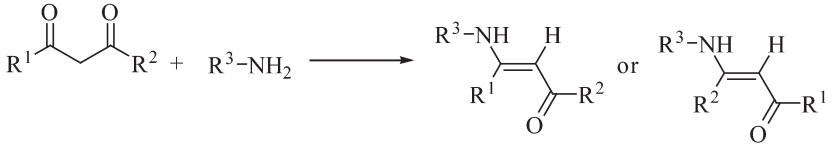
Initially, we employed 1-phenylbutane-1,3-dione (**1a**) with *p*-toluidine (**2a**) as the model reaction and tried to establish an effective reaction system for the reaction. These results are shown in Table 1. Among the Lewis acids tested, $FeCl_3$ showed the highest activity for this transformation (entries 2–4) and the reaction would not work in the absence of Lewis acid (entry 1). Among the various solvents examined, 1,2-dichloroethane (DCE) was successful (entries 5–8). When the reaction proceeded under refluxing condition using DCE as the solvent, the effect on increasing the yield was not obvious. Surprisingly, the reaction was found to proceed smoothly and led to the desired product in 88% yield when 0.2 equiv. *p*-toluenesulfonic acid (PTSA) was added to the reaction (entry 9). An increase in the amount of PTSA to 0.5 equiv., led to the same yield of the product (entry 11). The reaction also resulted in 58% yield in the absence of $FeCl_3$ (entry 10). A survey of the reaction time and the amount of $FeCl_3$ indicated that 2 h was practical and 0.1 equiv. of catalyst was the optimum amount (entries 9, 12–15). After screening, the optimal reaction conditions were obtained; that is, the mixture of 1-phenylbutane-1,3-dione (**1a**) with *p*-toluidine (**2a**), 0.1 equiv. of $FeCl_3$ and 0.2 equiv. of PTSA reacted in DCE at 80 °C for 2 h (Table 1, entry 9).

To explore the substrate scope and limitations of this reaction, a range of substrates were examined under the optimised reaction conditions. These results are summarised in Table 2. Treatment of 1-phenylbutane-1,3-dione (**1a**) with various amines **2** furnished the corresponding β-N-substituted (*E*)-Enaminones **3** in good to excellent isolated yields (**3aa–af**). As shown in Scheme 2, aromatic amines whether with electron-withdrawing groups or with electron-donating groups are all suitable for this protocol. Besides, the reaction appears quite tolerant with respect to the electronic contribution of the substituent on the benzene ring of aromatic amines. For example, the reaction of naphthalen-1-amine (**2c**) and 4-methoxybenzenamine (**2d**) with 1-phenylbutane-1,3-dione (**1a**) led to (*E*)-4-(naphthalen-1-ylamino)-4-phenylbut-3-en-2-one (**3ac**) and (*E*)-4-(4-methoxyphenylamino)-4-phenylbut-3-en-2-one in excellent yield. Interestingly, the amines bearing an aliphatic substituent proved to be a suitable partner (**3ab**, **3af**).

To demonstrate the generality of this method, we next investigated the scope of this reaction to other 1,3-dicarbonyl compounds, such as ethyl 3-oxo-3-phenylpropanoate and 1,1,1-trifluoropentane-2,4-dione. The reactions of ethyl 3-oxo-3-phenylpropanoate with p-toluidine (**2a**), naphthalen-1-amine (**2c**) and *m*-toluidine (**2g**) all lead to the corresponding (*E*)-aminoacrylate in good yield with high region- and stereo-selectivity (**3ba**, **3bc**, **3bg**). Additionally, 1,1,1-trifluoropentane-2,4-dione was also a good partner of 1,3-dicarbonyl compounds (**3ca**, **3cc**, **3cg**). The experimental results suggested a mechanism through the formation of imine intermediate.



Scheme 2 The configuration of **3ad**.



Scheme 1 Synthesis of β-functionalised enamine derivatives.

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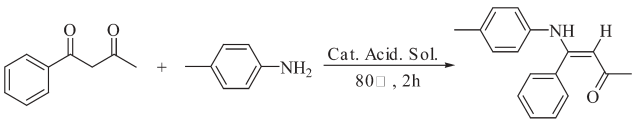
Table 1 Optimisation of reaction conditions[a]

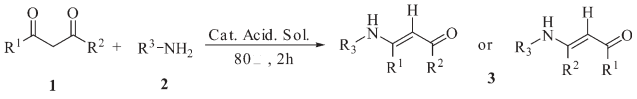


1a 2a 3aa

Entry	Cat. (0.1 equiv.)	Solvent	Yield[b]/%
1	—	Dioxane	none
2	$ZnCl_2$	Dioxane	52
3	$CuCl_2$	Dioxane	43
4	$FeCl_3$	Dioxane	74
5	$FeCl_3$	DMSO	67
6	$FeCl_3$	DMF	72
7	$FeCl_3$	Toluene	68
8	$FeCl_3$	DCE	79
9[c]	$FeCl_3$	DCE	88
10[c]	—	DCE	58
11[d]	$FeCl_3$	DCE	88
12[c,e]	$FeCl_3$	DCE	31
13[c,f]	$FeCl_3$	DCE	89
14[c,g]	$FeCl_3$	DCE	83
15[c,h]	$FeCl_3$	DCE	88

[a] **1a** (0.25 mmol), **2a** (0.25 mmol), solvent (4 mL); [b] GC yield; [c] PTSA (0.2 equiv.); [d] PTSA (0.5 equiv.); [e] reaction time: 0.5 h; [f] reaction time: 4 h; [g] $FeCl_3$ (0.05 equiv.); [h] $FeCl_3$ (0.2 equiv.).

Table 2 Synthesis of β-*N*-substituted (*E*)-enaminones and (*E*)-aminoacrylates[a]



1 2 3

1a (R^1=Me; R^2=Ph) **2a** =p-toluidine **2e** =pyridin-2-amine
1b (R^1=CO_2Et; R^2=Ph) **2b** =phenylmethanamine **2f** =hexan-1-amine
1c (R^1=Me; R^2=CF_3) **2c** =napthalen-1-amine **2g** =m-toluidine
2d =4-methoxybenzenamine

Entry	1	2	3	Yield/%[b]
1	**1a**	**2a**	**3aa**	86
2	**1a**	**2b**	**3ab**	82
3	**1a**	**2c**	**3ac**	75
4	**1a**	**2d**	**3ad**	85
5	**1a**	**2e**	**3ae**	80
6	**1a**	**2f**	**3af**	83
7	**1b**	**2a**	**3ba**	79
8	**1b**	**2c**	**3bc**	74
9	**1b**	**2g**	**3bg**	81
10	**1c**	**2a**	**3ca**	84
11	**1c**	**2c**	**3cc**	72
12	**1c**	**2g**	**3cg**	73

[a] **1** (1.0 mmol), **2** (1.0 mmol), DCE (4 mL), $FeCl_3$ (0.1 equiv.), PTSA (0.2 equiv.).
[b] Isolated yield

Unstability of the imine intermediate quickly isomerised to more stably conjugated enamine form (Scheme 3).

In summary, we have developed a facile and highly efficient condensation reaction to synthesise β-N-substituted (*E*)-enaminones and (*E*)-aminoacrylates with high region- and stereoselectivity via using the co-catalytic system of $FeCl_3$/PTSA. Further investigations concerning on the scope of this condensation reaction, applications are ongoing in our laboratory.

Experimental

All the reactions were carried out at 80 °C in a Schlenk tube equipped with magnetic stir bar. Solvents and all reagents were used as received. NMR spectra and GC–MS was obtained using an electron ionisation (EI) Agilent 6890N/5973 mass spectrometer. IR spectra were obtained with a Bruker Vector 22 spectrometer. Microanalysis was obtained using a Vario EL cube CHNOS elemental analyser. All the other chemicals were purchased from Aldrich Chemicals.

*Synthesis of (E)-4-(p-tolylamino)-4-phenylbut-3-en-2-one (***3aa***); general procedure*

$FeCl_3$ (16.2 mg, 0.1 mmol), *p*-toluenesulfonic acid (34.4 mg, 0.2 mmol), DCE (4 mL), 1-phenylbutane-1,3-dione (**1a**) (162 mg, 1.0 mmol) and *p*-toluidine (**2a**) (107 mg, 1.0 mmol) was added to a 10 mL Schlenk tube. The mixture was stirred at 80 °C for 2 h. The solution was directly subjected to isolation by PTLC (GF254), eluted with a 10:4 petroleum ether/ethyl acetate mixture, which furnished **3aa** (203.3 mg, 83%) as an orange oil.

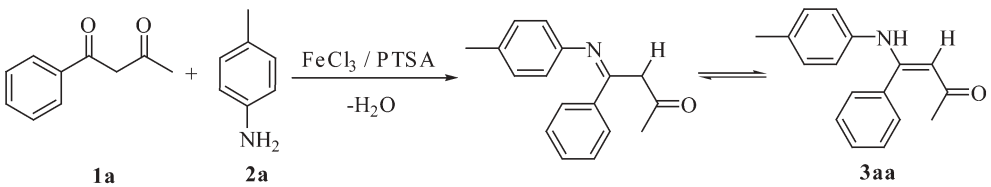
(E)-4-(p-Tolylamino)-4-phenylbut-3-en-2-one (**3aa**): Orange oil; ^{1}H NMR ($CDCl_3$, 400 Hz) δ 13.03 (s, 1H), 7.92 (d, *J* = 8.0 Hz, 2H), 7.52–7.49 (m, 3H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 5.86 (s, 1H), 2.34 (s, 3H), 2.09 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz) δ 188.3, 162.5, 140.0, 135.6, 130.7, 129.6, 128.1, 126.9, 124.7, 93.8, 20.8, 20.2; MS (EI) *m/z* (%): 250.95 (100.00); Anal. Calcd for $C_{17}H_{17}NO$: C, 81.24; H, 6.82. Found: C, 81.11; H, 6.94%.

(E)-4-(Benzylamino)-4-phenybut-3-en-2-one (**3ab**): Orange oil; IR ν_{max} (KBr): 3442, 2926, 1720, 1598, 1444, 1294, 1064, 800, 738, 698 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 Hz) δ 12.89 (b, 1H), 7.89 (d, *J* = 8.0 Hz, 2H), 7.43–7.29 (m, 8H), 6.17 (s, 1H), 4.64 (d, *J* = 4.0 Hz, 2H), 2.18 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz) δ 188.2, 161.2, 129.3, 128.7, 128.3, 127.8, 127.5, 94.8, 21.2; MS (EI) *m/z* (%): 91.05 (100.00), 251.00 (44.52); Anal. Calcd for $C_{17}H_{17}NO$: C, 81.24; H, 6.82. Found: C, 81.36; H, 6.81%.

(E)-4-(Naphthalen-1-ylamino)-4-phenylbut-3-en-2-one (**3ac**): Orange solid; m.p. 130–131°C; IR ν_{max} (KBr): 3448, 3053, 1587, 1552, 1425, 1286, 1219, 925, 786, 692 cm^{-1}; $^{-1}$H NMR ($CDCl_3$, 400 Hz) δ 13.6 (s, 1H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.79 (d, *J* = 8.0 Hz, 2H), 7.57–7.53 (m, 2H), 7.51–7.44 (m, 6H), 7.31 (d, *J* = 8.0 Hz, 1H), 6.02 (s, 1H), 2.02 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz) δ 189.2, 164.1, 140.2, 134.4, 130.1, 128.5, 127.4, 125.5, 123.0, 94.3, 20.4; MS (EI) *m/z* (%): 286.93 (100.00); Anal. Calcd for $C_{20}H_{17}NO$; C, 83.59; H, 5.96. Found: C, 83.66; H, 6.10%.

(E)-4-(4-Methoxyphenlamino)-4-phenylbut-3-en-2-one (**3ad**): Brown solid; m.p. 111–113 °C; IR ν_{max} (KBr): 3437, 1610, 1508, 1433, 1328, 1244, 1184, 1107, 1028, 831, 759, 705 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 Hz) δ 12.91 (s, 1H), 7.91–7.88 (q, 2H), 7.44–7.41 (m, 3H), 7.11–7.07 (t, 2H), 7.89–7.86 (t, 2H), 5.86 (s, 1H), 3.80 (s, 3H), 2.05 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz) δ 188.6, 163.4, 158.0, 140.3, 131.0, 128.5, 127.2, 126.8, 114.5, 93.7, 55.7, 20.5; MS (EI) *m/z* (%): 105.05 (100.00), 267.05 (83.36); Anal. Calcd for $C_{17}H_{17}NO_2$; C, 76.38; H, 6.41. Found: C, 76.81; H, 6.62%.

(E)-4-Phenyl-4-(pyridin-2-ylamino)but-3-en-3-one (**3ae**): Orange oil; IR ν_{max} (KBr): 3442, 2927, 1724, 1612, 1479, 1205, 1031, 856, 771, 694 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 Hz) δ 13.47 (s, 1H), 8.30 (d, 1H), 7.93 (d, 2H), 7.85–7.82 (m, 2H), 7.53–7.51 (m, 1H), 7.49–7.46 (m, 3H), 6.96–6.93 (t, 2H), 5.93 (s, 1H), 2.60 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz) δ 189.7, 161.4, 153.3, 148.3, 139.9,138.2, 131.5, 128.6, 127.5, 118.8, 115.4, 97.6, 23.1; MS (EI) *m/z* (%): 237.85 (100.00); Anal. Calcd for $C_{15}H_{14}N_2O$; C, 75.61; H, 5.92. Found: C, 75.66; H, 5.74%.



1a 2a 3aa

Scheme 3 Reaction.

(E)-4-(Hexylamino)-4-phenylbut-3-en-2-one (**3af**): Yellow oil; IR ν_{max} (KBr): 3410, 3062, 2931, 1708, 1597, 1444, 1307, 1078, 1022, 925, 725 cm^{-1}; ^1H NMR ($CDCl_3$, 400 Hz) δ 11.46 (s, 1H), 7.85–7.82 (m, 2H), 7.38–7.34 (m, 3H), 5.63 (s, 1H), 3.32–3.27 (q, 2H), 2.05 (s, 3H), 1.63–1.61 (m, 2H), 1.44–1.29 (m, 6H), 0.88–0.86 (t, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz) δ 188.1, 141.5, 130.6, 128.4, 127.1, 124.9, 93.1, 43.7, 31.7, 30.2, 26.8, 23.6, 19.7, 14.3; MS (EI) *m/z* (%): 105.05 (100.00), 245.10 (70.66); Anal. Calcd for $C_{16}H_{23}NO$; C, 78.32; H, 9.45. Found: C, 78.14; H, 9.55%.

(E)-Ethy 3-(p-tolylamino)-3-phenylacrylate (**3ba**):[37] Yellow solid; m.p. 69–71 °C; isolated yield 79%; ^1H NMR ($CDCl_3$, 400 Hz) δ 10.24 (s, 1H), 7.29–7.22 (m, 5H), 6.85 (d, *J* = 8.0 Hz, 2H), 6.54 (d, *J* = 8.0 Hz, 2H), 4.93 (s, 1H), 4.20–4.15 (q, *J* = 8.0 Hz, 2H), 2.17 (s, 3H), 1.30–1.27 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz) δ 170.4, 138.0, 129.6, 129.4, 129.0, 128.7, 128.6, 128.5, 122.6, 91.3, 59.5, 20.9, 14.8.

(E)-Ethyl 3-(naphthalen-1-ylamino)-3-phenylacrylate (**3bc**):[38] Yellow solid; m.p. 143–145 °C; isolated yield 74%; ^1H NMR ($CDCl_3$, 400 Hz) δ 10.66 (s, 1H), 8.32 (d, *J* = 8.0 Hz, 1H), 7.81–7.79 (d, *J* = 8.0 Hz, 1H), 7.56–7.47 (m, 4H), 7.29–7.16 (m, 4H), 7.04–7.02 (t, 1H), 6.49 (d, *J* = 8.0 Hz, 1H), 5.15 (s, 1H), 4.26–4.22 (q, *J* = 4.0 Hz, 2H), 1.34–1.32 (t, *J* = 4.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz) δ 170.7, 160.6, 136.5, 136.4, 134.3, 129.6, 128.5, 128.2, 126.6, 125.3, 124.2, 122.3, 121.4, 92.0, 59.7, 14.8.

(E)-Ethyl 3-(m-tolylamino)-3-phenylacrylate (**3bg**):[39] Yellow oil; isolated yield 81%; ^1H NMR ($CDCl_3$, 400 Hz) δ 10.52 (s, 1H), 7.30–7.22 (m, 5H), 6.91–6.88 (m, 1H), 6.69–6.67 (t, 1H), 7.37–6.34 (q, 1H), 4.95 (s, 1H), 4.20–4.17 (q, *J* = 4.0 Hz, 2H), 2.13 (s, 3H), 1.29–1.27 (t, *J* = 4.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz) δ 170.3, 159.3, 140.5, 138.7, 136.3, 128.7, 128.4, 123.0, 119.4, 91.1, 59.5, 21.5, 14.7.

(E)-4-(p-Tolylamino)-1,1,1-trifluoropent-3-en-2-one (**3ca**): Orange oil; ^1H NMR ($CDCl_3$, 400 Hz) δ 12.53 (s, 1H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.02 (d, *J* = 8.0 Hz, 2H), 5.50 (s, 1H), 2.35 (s, 3H), 2.07 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz) δ 176.8, 168.6, 137.9, 134.7, 130.4, 125.5, 119.4, 91.06, 21.4, 20.6; MS (EI) *m/z* (%): 174.05 (100.00), 243.00 (64.05); Anal. Calcd for $C_{12}H_{12}F_3NO$; C, 59.26; H, 4.97. Found: C, 59.33; H, 5.11.

(E)-4-(m-Tolylamino)-1,1,1-trifluoropent-3-en-2-one (**3cg**): Orange oil; IR ν_{max} (KBr): 3442, 2927, 1710, 1615, 1579, 1500, 1438, 1388, 1120, 1031, 887, 786, 694 cm^{-1}; ^{-1}H NMR ($CDCl_3$, 400 Hz) δ 12.54 (s, 1H),7.29–716 (m, 1H), 7.11 (d, *J* = 8.0 Hz, 1H), 6.96–6.94 (m, 2H), 5.51 (s, 1H), 2.36 (s, 3H), 2.09 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz) δ 176.5, 168.2, 139.9, 137.1, 129.5, 128.5, 126.1, 122.5, 116.8, 91.0, 21.5, 20.5; MS (EI) *m/z* (%): 174.10 (100.00), 243.00 (46.89); Anal. Calcd for $C_{12}H_{12}F_3NO$; C, 59.26; H, 4.97. Found: C, 59.11; H, 5.02%.

(E)-1,1,1-Trifluoro-4-(naphthalen-1-ylamino)pent-3-en-2-one (**3cc**): Brown solid; m.p. 80–81 °C; IR ν_{max} (KBr): 3442, 3061, 1675, 1579, 1427, 1382, 1246, 1120, 867, 771 cm^{-1}; ^{-1}H NMR ($CDCl_3$, 400 Hz) δ 12.78 (s, 1H), 7.92–7.85 (m, 3H), 7.58–7.49 (m, 3H), 7.32 (d, *J* = 8.0 Hz, 1H), 5.65 (s, 1H), 1.99 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 Hz) δ 169.7, 134.5, 133.4, 129.7, 128.8, 128.7, 127.8, 127.2, 125.4, 124.1, 122.4, 119.2, 91.1, 20.4; MS (EI) *m/z* (%): 278.95 (100.00); Anal. Calcd for $C_{15}H_{12}F_3NO$; C, 64.51; H, 4.33. Found: C, 64.30; H, 4.41%.

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