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Multicomponent preparation of highly functionalized piperidines using $FeCl_3.6H_2O$ as an efficient catalyst

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ABSTRACT

$FeCl_3.6H_2O$ was used as an efficient catalyst for the synthesis of highly functionalized piperidines via a one-pot five-component reaction of aromatic amines, aromatic aldehydes and *β*-keto esters in EtOH at room temperature. The remarkable advantages offered by this method are good yields, simple procedure, short reaction times, no need to column chromatography and easy work-up.

Keywords*: Multicomponent, Functionalized piperidine, Aldehyde, β-Keto ester.*

1. Introduction

Multicomponent [1,2] and domino [3,4] reactions allow the creation of several bonds in a single operation and are attracting increasing attention as one of the most powerful emerging synthetic tools [5,6]. Multicomponent reactions are defined as domino reactions involving at least three substrates and, consequently, constitute a subgroup of domino reactions [7]. The use of two component domino and multicomponent reactions in organic synthesis is increasing constantly. Such single-step reactions allow the synthesis of a wide range of complex molecules, including natural products and biologically active compounds such as pharmaceuticals and agrochemicals, in an economically favourable way by using processes that are reasonably simple [8-12].

The piperidines and their analogues are important heterocycles that are present in many naturally occurring alkaloids, biologically active synthetic molecules, and organic fine chemicals [13-15]. Some of them also act as pharmaceutical agents [16]. Compounds containing piperidine structural motif exhibit anti-hypertensive [17], antibacterial [18], antimalarial [19], anticonvulsant, and anti-inflammatory activities [20]. Furthermore, these compounds are intricately involved in the MAO based mechanism of Parkinson's disease [21,22] and as inhibitors of farnesyltransferase [23,24], and dihydroorotate dehydrogenase (Fig 1) [25]. In this respect, substituted piperidines have been identified as an important class of therapeutic agents in the treatment of influenza infection [26,27], cancer metastasis [28,29], viral infections including AIDS [30], and diabetes [31].

In recent years, the syntheses of functionalized piperidines were reported using plethora of reagents, such as a combination of L-proline / TFA [19], $InCl_3$ [32,33], tetrabutylammoniumtribromide (TBATB) [34], bromodimethylsulfonium bromide (BDMS) [35], cerium ammonium nitrate (CAN) [36], iodine [37], $ZrOCl_2.8H_2O$ [38], VCl_3 [39], $Bi(NO_3)_3.5H_2O$ [40], $BF_3.SiO_2$ [41], $LaCl_3.7H_2O$ [42], $NiCl_2.6H_2O$ [43], $Zn(HSO4)_2$ [44], $Mg(HSO4)_2$ [45] as a catalyst. However, some of these methods have drawbacks, such as long reaction times, unsatisfactory yields, the use of high temperature, expensive catalyst or difficult to prepared. Owing to the importance of piperidines from a pharmaceutical and biological point of view, there is still need to develop an efficient, mild reaction benign protocol for the synthesis of highly substituted piperidines. It is well known that $FeCl_3.6H_2O$ as a

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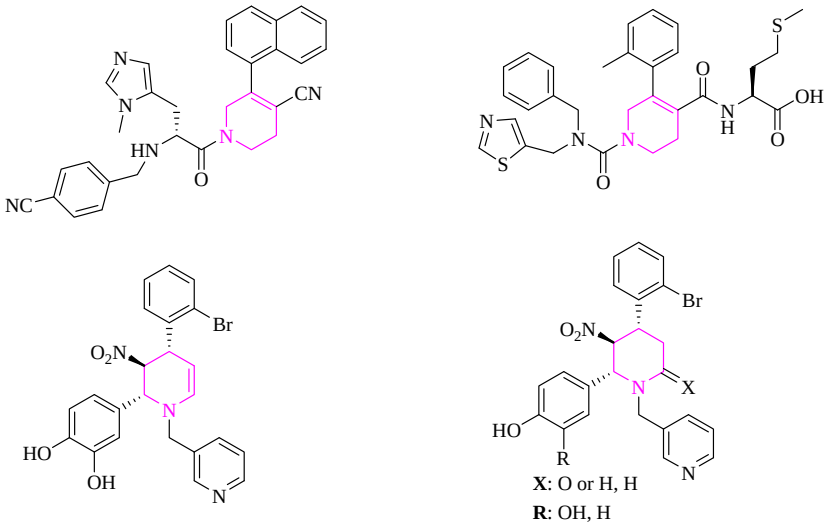


Fig 1. Farnesyltransferase active compounds containing piperidine framework [23,24].

Lewis acid catalyst have gained interesting attraction in recent years due to operational simplicity, efficient, low cost, ease of preparation and handling, more stable and economic consideration [46,47].

As a part of our current studies on development of efficient multi-component reactions for the preparation of interesting bioactive molecules [48-54], herein we report here an simple and efficient procedure for the synthesis of highly substituted piperidines via a one-pot five-component reaction between aromatic aldehydes, anilines and *β*-ketoesters in the presence of $FeCl_3.6H_2O$ as a catalyst (Scheme 1).

2. Experimental

2.1. General

Melting points were measured on an Electrothermal 9100 apparatus. The ^{1}HNMR spectra were recorded on a Bruker DRX-400 Avance instrument with $CDCl_3$ as solvent. All reagents were purchased from Merck (Darmastadt, Germany), Acros (Geel, Belgium) and Fluka (Buchs, Switzerland), and used without further purification.

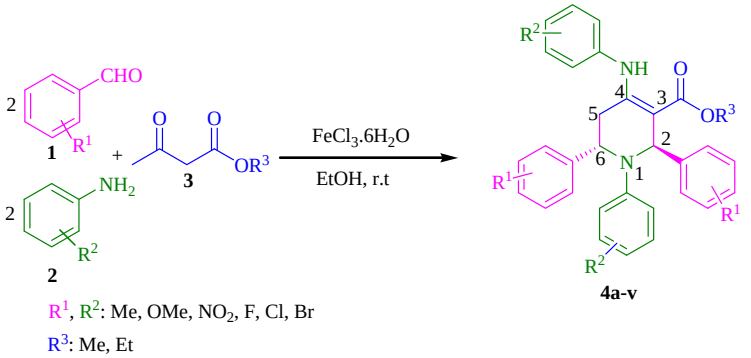
*2.2. General procedure for the synthesis of highly functionalized piperidine **4***

A solution of aromatic amine **2** (2.0 mmol) and β-ketoester **3** (1.0 mmol) in EtOH (4 mL) was stirred for 20 min in the presence of 0.30 mmol $FeCl_3.6H_2O$ at room temperature. Next, the aromatic aldehyde **1** (2.0 mmol) was added and the reaction mixture was stirred for the time indicated in Table 3. The progress of the reaction was monitored by thin-layer chromatography (TLC). After completion of the reaction, the thick precipitate was filtered off and washed with ethanol (3×2 mL) to give the pure product **4**.

Selected spectral data

*Compound (**4a**)*:

^{1}HNMR (400 MHz, $CDCl_3$): δ= 2.36, 2.38 (6H, 2s, $2ArCH_3$), 2.80 (1H, dd, *J*= 15.2, 2.4 Hz, H'-5), 2.91 (1H, dd, *J*= 14.0, 5.6 Hz, H"-5), 3.96 (3H, s, OCH_3), 5.15 (1H, d, *J*= 2.4 Hz, H-6), 6.34 (2H, d, *J*= 8.0 Hz, ArH), 6.44 (1H, s, H-2), 6.56 (2H, d, *J*= 8.0 Hz, ArH), 6.63 (1H, t, *J*= 7.2 Hz, ArH), 7.07 -7.25 (13H, m, ArH), 10.29 (1H, s, NH) ppm.



Scheme 1. Synthesis of highly functionalized piperidine **4a-v**.

*Compound (**4e**)*:

^{1}HNMR (400 MHz, $CDCl_3$): δ= 2.78 (1H, dd, *J*= 15.2, 2.4 Hz, H'-5), 2.87 (1H, dd, *J*= 15.2, 5.6 Hz, H"-5), 3.97 (3H, s, OCH_3), 5.14 (1H, d, *J*= 5.6 Hz, H-6), 6.40 (1H, s, H-2), 6.45 (2H, d, *J*= 7.2 Hz, ArH), 6.50 (2H, d, *J*= 8.0 Hz, ArH), 6.69 (1H, t, *J*= 7.2, ArH), 7.09-7.28 (13H, m, ArH), 10.30 (1H, s, NH).

*Compound (**4l**)*:

^{1}HNMR (400 MHz, $CDCl_3$): δ= 2.71 (1H, dd, *J*= 15.2, 2.4 Hz, H'-5), 2.86 (1H, dd, *J*= 15.2, 5.6 Hz, H"-5), 3.81, 3.83, 3.97 (9H, 3s, $3OCH_3$), 5.07 (1H, d, *J*= 3.6 Hz, H-6), 6.28 (2H, d, *J*= 8.0 Hz, ArH), 6.32 (1H, s, H-2), 6.46 (2H, d, *J*= 8.0 Hz, ArH), 6.83-7.20 (12H, m, ArH), 10.25 (1H, s, NH).

3. Results and Discussion

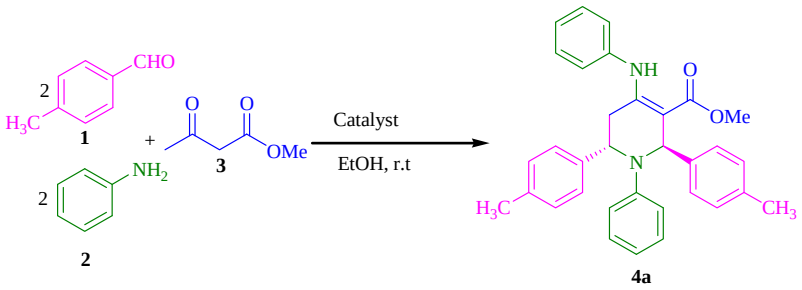
In the initial experiment, the one-pot five-component reaction between 4-methyl benzaldehyde, aniline and methyl acetoacetate was chosen as a model reaction and reacted under different experimental variants to optimize the reaction conditions (Table 1). We have compared $FeCl_3.6H_2O$ with other catalysts, the effect of $FeCl_3.6H_2O$ as catalysts in the reaction has been investigated. As indicated in Table 1, the reaction could be progressed very efficiently in the presence of $FeCl_3.6H_2O$ in ethanol. It is noteworthy that no product was obtained in the absence of catalyst even after 48 h (Table 1, Entry 7), which indicated that the catalyst's presence is necessary for this transformation.

The effect of solvents and catalytic amount of $FeCl_3.6H_2O$ in the reaction has been investigated (Table 2). The reaction could be progressed very efficiently in EtOH in the presence of 0.30 mmol of catalyst at ambient temperature by considering environmental aspects (Table 2, Entry 7). EtOH has been chosen as a green and suitable solvent for the synthesis. When the reaction was carried out under solvent-free conditions, the product was obtained in a moderate yield (35%) that may be due to lack of effective interaction of reactants with the catalyst in the absence of solvent (Table 2 Entry 13). The increasing of catalyst loading to 0.35 mmol had no improving effect on the yield of product (Table 2, Entry 8).

The procedure is very simple and clean. Products are separated from the reaction media with a simple filtration and no more purification is needed. EtOH use as a green solvent increases the environment-friendly aspects of the reaction.

To explore the scope and limitations of this reaction, various substituted benzaldehydes, anilines, and methyl or ethyl acetoacetate were examined under the optimized conditions and the results are summarized in Table 3. Benzaldehyde with electron-deficient or electron-releasing groups reacts efficiently with aniline or substituted aniline to give the corresponding piperidines in high yields. Various substituents on the benzene ring such as OMe, Me, NO_2, F, Cl, and Br were tolerated during the reaction. In all cases, the reaction proceeded to afford piperidine derivatives in good yields.

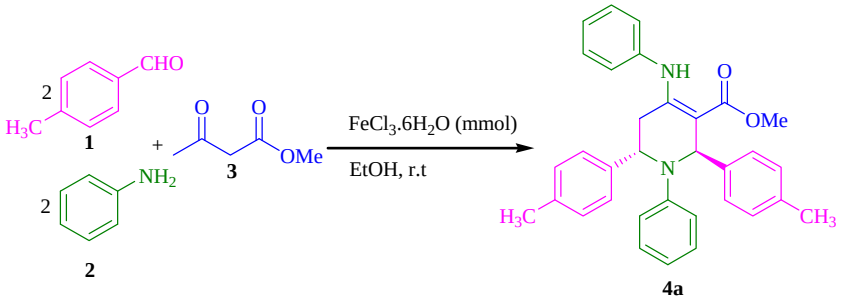
Table 1. The effect of different catalyst on the synthesis of piperdines[a]



Entry	Catalyst (mmol)	Time (h)	Isolated Yield (%)
1	K_2HPO_4 (0.10)	48	-
2	MoO_3 (0.10)	48	-
3	Succinic acid (0.10)	24	38
4	Tannic acid (0.10)	48	-
5	$PrCl_3.6H_2O$ (0.10)	18	45
6	**$FeCl_3.6H_2O$ (0.10)**	**15**	**65**
7	No Catalyst	48	-

[a]Reaction conditions: 4-methyl benzaldehyde (2.0 mmol), aniline (2.0 mmol) and methyl acetoactate (1.0 mmol) in EtOH (4 mL).

Table 2. Effect of solvents and catalytic amount of $FeCl_3.6H_2O$ for the synthesis of functionalized piperidines[a]



Entry	Solvent	Catalyst (mmol)	Time (h)	Isolated Yield (%)
1	EtOH	0.02	24	37
2	EtOH	0.05	20	54
3	EtOH	0.10	15	65
4	EtOH	0.15	11	68
5	EtOH	0.20	9	72
6	EtOH	0.25	8	78
7	**EtOH**	**0.30**	**6**	**89**
8	EtOH	0.35	6	89
9	H_2O	0.30	24	35
10	CH_3CN	0.30	9	60
11	MeOH	0.30	7	83
12	H_2O/EtOH	0.30	15	47
13	-	0.30	20	35

[a]Reaction conditions: 4-methyl benzaldehyde (2.0 mmol), aniline (2.0 mmol), methyl acetoactate (1.0 mmol) and solvent (4 mL).

The structures of all compounds were characterized by comparison of their IR and NMR spectra with authentic samples. Also, the relative stereochemistry of these piperidines has been confirmed by single X-ray crystallography analysis in previously reported literature [32-38, 40-41, 49], and the relative stereochemistry of the products in the present work was proved by comparison of spectroscopic data of some products with those authentic samples.

The formation of piperidines through a Knoevenagel-type intermediate followed by [4+2] aza-Diels-Alder reaction has been reported in the literature [19, 35, 38-39, 42, 50].

To show the merit of the present work in comparison with reported results in the literature, we compared results of $FeCl_3.6H_2O$ with L-proline/THF [19], $InCl_3$ [32], tetrabutylammonium tribromide (TBATB) [34], bromodimethylsulfonium bromide (BDMS) [35], cerium ammonium nitrate (CAN) [36], iodine [37], $ZrOCl_2.8H_2O$ [38], VCl_3 [39], $Bi(NO_3)_3.5H_2O$ [40], and *p*-TsOH.H_2O [48], in the synthesis highly functionalized piperidine derivatives. As shown in Table 4, $FeCl_3.6H_2O$ can act as effective catalyst with respect to reaction times and yields of products.

4. Conclusions

We have developed a simple and efficient method for the synthesis of highly substituted piperidines by one-pot multi-component reactions under mild conditions using $FeCl_3.6H_2O$ as the catalyst in ethanol. This reaction can be employed as an efficient approach for the preparation of synthetically and pharmaceutically important piperidine systems. The one-pot reaction has some important advantages such as the easy work-up procedure, simple and readily available precursors, high atom efficiency, clean reaction profiles, inexpensive catalyst and good to high yields.

Acknowledgment

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Table 3. The synthesis of highly substituted piperidine **4a**-**4v**.

Entry	R^1	R^2	R^3	Product	Time (h)	Yield (%)[a]	m.p. (°C)[b]		Ref.
							Found	reported	
1	4-Me	H	Me	**4a**	6	89	214-216	215-217	[37]
2	4-Me	H	Et	**4b**	6	83	227-229	228-231	[37]
3	4-OMe	H	Me	**4c**	8	74	186-188	187-188	[35]
4	4-OMe	H	Et	**4d**	8	82	165-167	166-168	[48]
5	4-Cl	H	Me	**4e**	12	80	188-190	189-191	[35]
6	3-NO_2	H	Me	**4f**	14	55	181-182	180-181	[54]
7	4-NO_2	H	Me	**4g**	8	80	237-239	239-241	[37]
8	H	H	Me	**4h**	6	85	169-170	169-171	[35]
9	H	H	Et	**4i**	7	75	172-174	174-175	[35]
10	H	4-OMe	Et	**4j**	8	90	179-181	179-181	[48]
11	H	4-Cl	Et	**4k**	14	70	198-200	197-198	[54]
12	4-OMe	4-Cl	Me	**4l**	10	86	192-195	194-195	[36]
13	4-Me	4-Br	Me	**4m**	5	68	228-231	230-232	[37]
14	4-Me	4-OMe	Me	**4n**	8	90	224-226	225-226	[35]
15	4-Me	4-OMe	Et	**4o**	7	84	219-222	221-224	[36]
16	4-Me	4-Me	Me	**4p**	6	81	207-209	206-208	[37]
17	4-Me	4-F	Et	**4q**	6	90	181-183	183-185	[48]
18	4-Me	3,4-di-Cl	Et	**4r**	18	71	172-175	173-175	[48]
19	4-NO_2	H	Et	**4s**	9	66	246-248	247-250	[36]
20	H	4-Br	Et	**4t**	7	77	197-199	196-198	[49]
21	4-OMe	4-Br	Me	**4u**	6	69	175-177	178	[19]
22	4-NO_2	4-OMe	Me	**4v**	7	60	197-200	198-199	[38]

[a]Isolated yield.

[b]All known products reported previously in the literature were characterized by comparison of IR and NMR spectra with those of authentic samples.

Table 4. Comparison of $FeCl_3.6H_2O$ with previously reported catalyst for the synthesis of highly functionalized piperidine **4a**.

Catalyst/	Conditions	Time (h)	Yield (%)	Ref.
L-proline	THF/CH_3CN, 20-30 ˚C	—	—	[19]
$InCl_3$	CH_3CN, r.t.	24	50	[32]
TBATB	EtOH, r.t.	3	80	[34]
BDMS	CH_3CN, r.t.	10	78	[35]
CAN	CH_3CN, r.t.	22	85	[36]
I_2	MeOH, r.t.	8	84	[37]
$ZrOCl_2.8H_2O$	EtOH, reflux	—	—	[38]
VCl_3	EtOH, r.t.	8	81	[39]
$Bi(NO_3)_3.5H_2O$	EtOH, r.t.	18	73	[40]
p-TsOH.H_2O	EtOH, r.t.	7	89	[45]
$FeCl_3.6H_2O$	EtOH, r.t.	6	89	This work

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