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Bis-amino methylation for the synthesis of spiro-fused piperidines using iron(III) trifluroacetate in aqueous micellar medium

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Abstract An environmentally benign, multicomponent integrated chemical process has been developed for the synthesis of 3,5-dispirosubstituted piperidines by cyclo-condensation reaction of amines, formaldehyde, and dimedone using iron(III) trifluroacetate [$Fe(F_3CCO_2)_3$] Lewis acid in aqueous micellar medium at ambient temperature (25–30 °C). The heterogeneous solid acid catalyst conveniently promotes this double amino methylation reaction in which six molecules condense in one pot to form six new covalent bonds. The synthesized 3,5-dispirosubstituted piperidines have been screened for their in vitro antibacterial activity using agar well method. Many of these compounds showed satisfactory antibacterial activity as compared to standard drugs against all the bacteria tested.

Keywords Multicomponent reactions · Iron(III) trifluroacetate · 3,5-Dispirosubstituted piperidines · Aqueous SDS micellar medium

Introduction

Multi-component reactions (MCRs) become one of the fundamental aspects of "Green Chemistry" since, it involves construction of novel and structurally important complex molecules. It combines several starting materials in a single pot ensuring high atom economy, good yields with low costs, minimizing waste, labor, and energy, short reaction time, simple work-up, and purification of products [1]. Therefore, MCRs synthetic strategy are currently very popular in organic, medicinal, and combinatorial chemistry for rapid and efficient generation of different biologically active compounds [2].

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It is well known that spiro-fused piperidine represents one of the most ubiquitous heterocyclic motifs and is present in many naturally occurring alkaloids, animal toxins, biologically active synthetic molecules, and organic fine chemicals [3–5]. These heterocyclic scaffolds possess broad biological activities such as anti-hypertensive, anti-convulsant, anti-inflammatory, anti-HIV-1, analgesic, leishmanicidal, antimicrobial, and cytotoxic activities [6–12]. The neurotoxic 2-spiro-fused piperidines were isolated from the neotropical poisonous frog [13–16] and their 3-spirofused analogues alkaloids such as sibirine, nitramine, isonitramine, and nitrabirine were isolated from plants of the genus *Nitraria* [17–19].

Conventionally, 3,5-dispirosubstituted piperidines are synthesized by using several modified procedures such as $FeCl_3$ in DCM [20], Fe_3O_4 nanoparticles [21], silica supported tungstic acid in DCM [22], $In(OTf)_3$ in ethanol [23], and in ethanol solvent only [24]. Although some of these reported methods afford good to high yields of the corresponding products, many of these methods suffer from various disadvantages such as long reaction time, moisture sensitivity, high cost, and toxic reagents. Hence, it is necessary to develop an efficient and convenient method to construct these biologically active heterocyclic compounds.

The classical Lewis acids such as $FeCl_3$, $AlCl_3$, BF_3, $ZnCl_2$, $TiCl_4$, and $SnCl_4$ used in organic synthesis are susceptible to fast hydrolysis by means of atmospheric moisture and accordingly should be treated strictly in dry conditions to avoid the loss of catalytic activity. Quite often, these acids are required in stoichiometric amounts, are not reusable, and lead to secondary reactions, making the reaction work-up and product isolation process tedious. Because of the increasing awareness of environmental problems in chemical research and industry, the search for a more efficient Lewis acid catalyst still continues.

Iron trifluroacetate $[Fe(CF_3CO_2)_3]$ is a non-hygroscopic, non-corrosive, stable, commercially available Lewis acid with high catalytic activity, economic viability, and easy recoverability from water. It has been used as a catalyst for solvolytic and non-solvolytic nucleophilic ring opening of epoxides [25], O-silylation of α-hydroxyphosphonates, alcohols, and phenols [26], synthesis of 3,4-dihydropyrimidinones and 1,4-dihydropyridines [27].

From a viewpoint of ecological advantage and greenness of water, it is of considerable interest to perform organic reactions in water because water is abundant in nature, has virtually no cost, and is safest among the available solvents, thus leading to environmentally benign chemical processes [28]. Recently, some attempts to achieve organic reactions in aqueous media have been performed and consequently some successful examples have appeared in the literature [29, 30]. The use of water in organic reactions has serious limitations. Most organic compounds are immiscible in water and many are reactive, while catalysts, reagents, and products are decomposed or deactivated in aqueous media. To overcome these drawbacks, surface active compounds (surfactants) can be used successfully in the water, which solubilize organic materials or form emulsions with them in water [31]. The solubilization of water-insoluble reactants and products inside the micelles results not only in high concentration within the small volume, but also in different orientations of the soluble molecules that influence the reaction

mechanism, resulting in remarkable differences in the reaction rate and selectivity that would be observed in a homogeneous system [32].

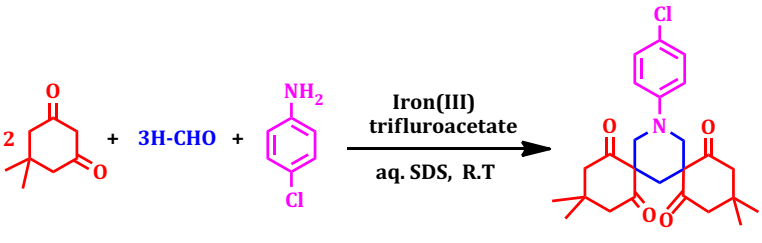
Considering the significance of anionic sodium dodecyl sulphonate surfactant (SDS) [33] and in continuation of our research work to develop new and convenient synthetic protocols for the construction of bioactive heterocycles [34], we aim to investigate the efficiency of $[Fe(F_3CCO_2)_3]$ solid Lewis acid for the synthesis of 3,5-dispirosubstituted piperidines in aqueous SDS micellar medium.

Results and discussion

Initially, to establish the feasibility of the strategy and to optimize the reaction conditions, reaction of dimedone, formaldehyde, and 4-chloroaniline as a model substrate was investigated (Scheme 1).

The reaction was carried out in 10 mol% aqueous SDS micellar medium, only 30 % of resulting product obtained after 10 h at room temperature (Table 1, entry 1). To improve the yield, a representative selection of iron(III) metal salts including $FeCl_3$, $Fe(NO_3)_2$, $Fe(OH)_3$, and $[Fe(CF_3CO_2)_3]$ were tested in aq. SDS micellar medium. Among the tested metal salts, $[Fe(CF_3CO_2)_3]$ was found to be a more efficient and effective Lewis acid for this bis-amino methylation (Table 1, entries 2–5). The promising activity of iron(III) trifluoroacetate $[Fe(CF_3CO_2)_3]$ as compared to other metal complex salts can be explained on the basis of the spectrochemical series and hard-soft acids and bases (HSAB) principle [23]. Possibly, the trifluoroacetate anion coordinates with enol form of dimedone better than other ions such as Cl^-, NO_3^-, and OH^- ions, which enhances the rate of reaction.

With $[Fe(CF_3CO_2)_3]$ as catalyst, we next investigated the effect of solvents and found that aq. SDS micellar medium was superior to organic solvents such as DCM, $CHCl_3$, and acetonitrile (Table 1, entries 6–8). Moreover, in protogenic solvents such as water and ethanol, protonation of amines prior to attack may slow the nucleophilicity of the amine moiety, thus resulting in lower yields (Table 1, entries 9, 10). Performing the reaction with a higher catalyst loading 10 mol% $[Fe(CF_3CO_2)_3]$ catalyst had no significant effect on yield. However, if the amount of the catalyst was reduced to 2 and 1 mol%, the product yield was reduced to 76 and 72 %, respectively (Table 1, entries 11, 12). To our delight, the reaction worked



Scheme 1 Synthesis of 3,5-dispirosubstituted 4-chloro-phenyl piperidine

Table 1 Optimization of reaction conditions for the synthesis of 3,5-dispirosubstituted 4-chloro-phenyl piperidine

Entry	Solvent	Catalyst	Mol (%)	Time (h)	Yield[a] (%)
1	Aq. SDS	–	10	10	30
2	Aq. SDS	$FeCl_3$	5	5.5	80
3	Aq. SDS	$Fe(NO_3)_3$	5	6.0	70
4	Aq. SDS	$Fe(OH)_3$	5	6.5	68
5	Aq. SDS	$[Fe(CF_3CO_2)_3]$	5	4.5	86
6	CH_2Cl_2	$[Fe(CF_3CO_2)_3]$	5	5.5	80
7	$CHCl_3$	$[Fe(CF_3CO_2)_3]$	5	5.5	78
8	CH_3CN	$[Fe(CF_3CO_2)_3]$	5	6.0	75
9	C_2H_5OH	$[Fe(CF_3CO_2)_3]$	5	6.0	75
10	Water	$[Fe(CF_3CO_2)_3]$	5	6.5	68
11	Aq. SDS	$[Fe(CF_3CO_2)_3]$	2	5.5	76
12	Aq. SDS	$[Fe(CF_3CO_2)_3]$	1	5.5	72

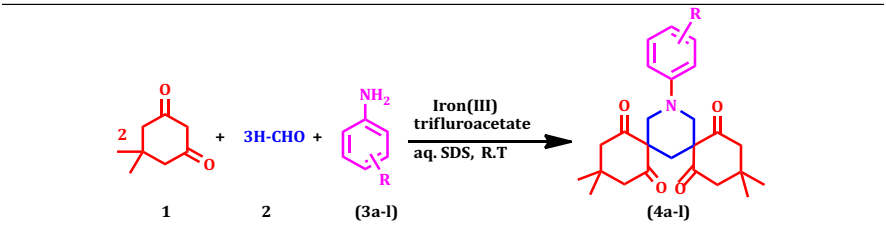
Reaction conditions: 4-chloroaniline (1 mmol), dimedone (2 mmol), formaldehyde (3 mmol) stirred at R.T.

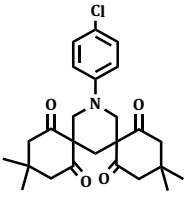
very well in the presence of 10 mol% SDS under the influence of 5 mol% iron(III) trifluroacetate catalyst in water that led to the desired condensed adduct in 86 % isolated yield after 4.5 h (Table 1, entry 5). It seems that the Lewis acid part of the iron(III) trifluroacetate catalyst activates the substrate molecules and the surfactant part of SDS increases the solubility of the substrates in water at the same time. Because of this synergistic effect, a drastic enhancement of the reaction rate and yield of the product was observed.

With these encouraging results in hand, we turned to explore the scope of the reaction using different amines as substrate under the optimized reaction conditions (Table 2). It was observed that aromatic amines with electron-donating as well as electron-withdrawing groups reacted successfully to furnish the final product in good to excellent yields, while the presence of a strong electron-withdrawing group such as $–NO_2$ on the phenyl ring results in lower yield (Table 2, **4f**). *o*-nitro aniline does not yield a spiro product due to relatively weak nucleophilicity and, therefore, decreased activity in the Mannich step of the reaction (Table 2, **4k**). Aliphatic amines such as n-butyl amine afforded a corresponding product in 82 % (Table 2, **4i**), but cyclohexyl amine does not yield any spiro adduct after 24 h due to its ring strain effect (Table 2, **4j**). It should also be noted that sterically hindered 1-naphthylamine (Table 2, **4l**) did not give the corresponding spiropiperidine.

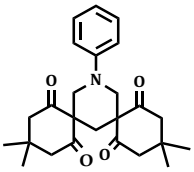
From the mechanistic point of view, a plausible mechanism for the formation of target compound is outlined in (Scheme 2) [21]. The spirocyclization looks to proceed as a domino sequence of Knoevenagel, Michael, and double Mannich reactions. The well-known reaction of dimedone with formaldehyde leads to the formation of the standard dimedone–formaldehyde adduct. This undergoes two consecutive Mannich reactions with aniline to produce the bis-spiro piperidine. Here

Table 2 Synthesis of 3,5-dispirosubstituted piperidines in aqueous SDS medium (**4a–l**)

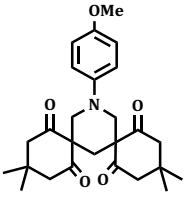




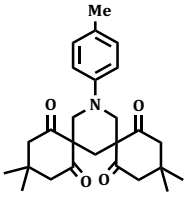
(4a, yield: 86 %)
(mp:190-192^{0}C)



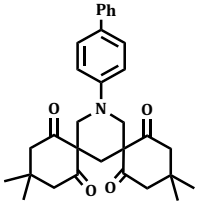
(4b, yield: 88 %)
(mp:195-198^{0}C)



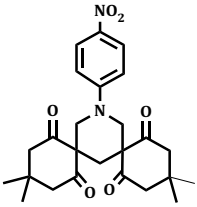
(4c, yield: 88 %)
(mp:202-204^{0}C)



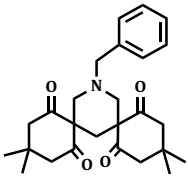
(4d, yield: 87 %)
(mp:198-200^{0}C)



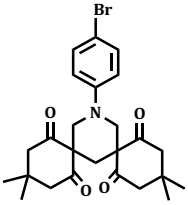
(4e, yield: 81 %)
(mp:188-190^{0}C)



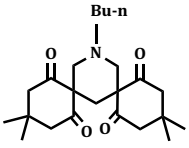
(4f, yield: 75 %)
(mp:220-222^{0}C)



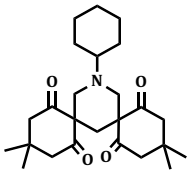
(4g, yield: 80 %)
(mp:196-198^{0}C)



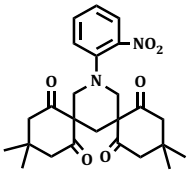
(4h, yield: 82 %)
(mp:200-202^{0}C)



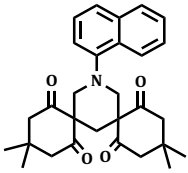
(4i, yield: 82 %)
(m.p: 210-212^{0}C)



(4j, n.r.)



(4k, n.r.)

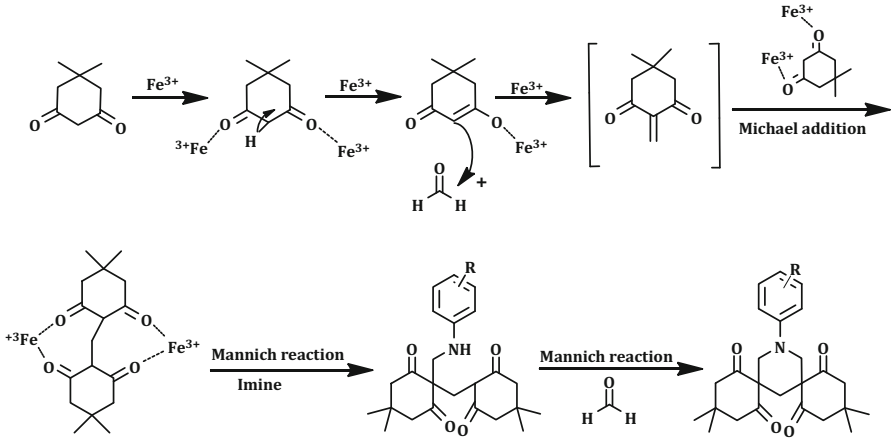


(4l, n.r.)

Reaction conditions: Formaldehyde (3 mmol), amines (1 mmol), dimedone (2 mmol), 5 mol % Iron (III)-trifluroacetate, 10 mol % aq. SDS micellar medium at R.T
n.r.: No Reaction

the acidic nature of iron(III) trifluroacetate facilitates both the enolization steps of the dimedone.

Recycling of the catalyst is important for the large-scale operation and industrial point of view. To check the possibility of the catalyst recycling, reaction of 4-chloroaniline, formaldehyde, and dimedone in the presence of 5 mol%



Scheme 2 Plausible mechanism of 3,5-dispirosubstituted piperidine ring formation

$[Fe(CF_3CO_2)_3]$ in aq. SDS micellar medium was studied. In the model reaction, after product isolation by simple filtration, the filtrate containing catalyst was further applied by taking the same reactants in the aqueous phase to perform five successive catalytic runs. The results in Table 3 shows that the catalytic system could be used five times with only a slight decrease in the catalytic activity.

The synthesized 3,5-dispirosubstituted piperidines were screened for their in vitro anti-bacterial activity against the growth of Gram negative bacteria and Gram positive bacteria by using agar well diffusion and agar broth. As shown in the Tables 4 and 5, the tested compounds showed moderate antibacterial activity compared to standard drugs against each microorganism.

On the basis of zone of inhibition and MIC test against tested bacterial pathogens, compound **4c** shows good antibacterial activity against both Gram positive and Gram negative bacterial pathogens at 30 μg/mL. Also, compound **4g** possess good activity against Gram negative *S. aureus* at 30 μg/mL. The remaining compounds possess slightly moderate activity compared to the standard drug ampicillin.

The reason for different sensitivities between Gram positive and Gram negative bacteria could be ascribed to the morphological difference between these microorganisms. The Gram positive bacteria had been found to be more susceptible since they have only an outer peptidoglycan layer, which is not an effective permeability barrier [35]. Gram negative bacteria have an outer phospholipidic membrane carrying the structural lipopolysaccharide components. This makes the cell wall

Table 3 Reusability of catalyst in the model reaction

Entry	No. of cycles	Yield (%)[a]
1	1	86
2	2	84
3	3	81
4	4	80
5	5	79

[a] Yields are related to isolated pure products

Table 4 Antibacterial activity of 3,5-dispirosubstituted piperidine derivatives

Entry	Compound	Zone of inhibition (mm)			
		S. aureus		*E. coli*	
		50 (μg/mL)	100 (μg/mL)	50 (μg/mL)	100 (μg/mL)
1	**4a**	2[a]	5	1	3
2	**4b**	2	4	–	–
3	**4c**	3	7	4	9
4	**4d**	1	3	1	4
5	**4e**	–	1	–	–
6	**4f**	2	4	2	4
7	**4g**	4	9	–	–
8	Standard amphicilin	22	20	23	20
9	Solvent (ethanol)	–	–	–	–
10	Control	–	–	–	–

[a] Zone inhibition values are given in millimeters, '–' no inhibition

Table 5 MIC values of compounds against Gram positive and Gram negative bacteria

Entry	Compound	*S. aureus* MIC (μg/mL)	*E. coli* MIC (μg/mL)
1	**4a**	40	50
2	**4b**	40	–
3	**4c**	30	30
4	**4d**	50	50
5	**4e**	100	–
6	**4f**	40	40
7	**4g**	30	–
8	Standard amphicilin	10	10
9	Control	–	–

'–' no inhibition

impermeable to lipophilic solutes, while porins constitute a selective barrier to the hydrophilic solutes with an exclusion limit of about 600 Da [36].

Experimental

General

Solvents and reagents were of AR grade commercially sourced from Sigma Aldrich and used without further purification. Melting points were determined in an open capillary and are uncorrected. Infrared spectra were measured on a Perkin Elmer FT-IR spectrophotometer. The samples were examined as KBr discs ~5 %w/w.

NMR spectra were recorded on a Bruker AC (300 MHz for ^{1}H NMR and 75 MHz for ^{13}C NMR) spectrometer using $CDCl_3$ and DMSO as solvents.

General procedure for the synthesis of bis-spiropiperidines (4a-l)

In a round bottom flask, a mixture of dimedone (2 mmol), formaldehyde (3 mmol), aniline (1 mmol), iron(III) trifluroacetate (5 mol%), and SDS surfactant (10 mol%) in water (5 mL) was stirred at R.T. The progress of reaction was monitored by thin layer chromatography. After the completion of the reaction, the formed product was filtered and recrystallized from ethanol to afford the pure product. Identity of the synthesized products was confirmed by comparing the physical and spectral data (MS, ^{1}H NMR, ^{13}C NMR) with those of the compounds reported in literature.

Agar well method for antibacterial activity of synthesized compounds

Antibacterial activity was tested on nutrient agar plates using agar well diffusion [37]. The organisms used in the present study were obtained from the laboratory stock. The subculture was prepared by pouring 15–20 mL of molten media into each sterile nutrient broth plate. The incubated plates were allowed to stand for 15 min. Using a sterile cork borer, the bores are made and solutions of synthesized compounds were prepared at different concentrations (μg/mL), and transferred into the bores. The control was run under similar conditions to get the activity of blanks. The plates were incubated at temperature of 37 °C. Antimicrobial activity was evaluated after 24 h by measuring the diameter (mm) of zone of inhibition.

Minimum inhibitory concentration assay

The minimum inhibition concentration assay was performed for the compounds at 10, 20, 30, 40, 50, 60, 70, 80, 90, and 100 μg/mL concentrations. A set of tubes with multiple concentrations of compounds was prepared in growth medium (agar broth). The tubes were then inoculated with the microorganisms, incubated for 24 h, and examined for growth of bacteria. Broth tubes that appear turbid are indicative of bacterial growth, while tubes that remain clear indicate no growth. Growth seems to diminish as the concentration of some compounds increase and eventually appeared to reduce at higher concentrations. The minimum concentration at which no growth was observed was taken as the MIC value.

Physical and spectral data of representative compounds

*Compound (**4a**)*

mp: 190–192 °C (lit. 198–200 °C [21]); ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 1.00 (12H, s, CH_3), 2.51 (2H, s, CH_2), 2.70 (4H, d, $J = 13.5$ Hz, $COCH_2$), 2.92 (4H, d, $J = 13.5$ Hz, $COCH_2$), 3.54 (4H, s, NCH_2), 7.10 (2H, d, $J = 8$ Hz, ArH), 7.31(2H, d, $J = 8$ Hz, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 28.2, 28.4, 30.7, 32.2, 51.2, 54.2, 65.4, 113.4, 120.4, 131.2, 150.4, 205.6; MS (*m*/*z*): 443(M^+).

*Compound (**4b**)*

mp: 195–198 °C (lit. 190–192 °C [22]); ^{1}H NMR (300 MHz,$CDCl_3$ δ ppm): 1.00 (12H, s, CH_3), 2.31 (2H, s, CH_2), 2.52 (4H, d, J = 13.5 Hz, $COCH_2$), 2.83 (4H, d, $COCH_2$), 3.69 (4H, s, NCH_2), 7.00–7.31 (5H, m, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 28.12, 28.21,30.55, 32.38, 51.20, 65.73, 118.80, 121.60, 129.20, 151.90, 205.80; MS (*m*/*z*): 409 (M^+).

*Compound (**4d**)*

mp: 198–200 °C (lit. 196–198 °C [20]); ^{1}H NMR (300 MHz,$CDCl_3$ δ ppm): 1.00 (12H, s, CH_3), 2.01 (3H, s, CH_3), 2.41 (2H, s, CH_2), 2.68 (4H, d, J = 13.5 Hz, $COCH_2$), 2.87 (4H, d, J = 13.5 Hz, $COCH_2$), 3.51 (4H, s, NCH_2), 7.22 (2H, d, J = 8.1 Hz, ArH), 7.43(2H, d, J = 8.1 ArH); ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 20.25, 28.55, 28.92, 30.71, 32.35, 51.52, 55.61, 66.12,119.62, 129.35, 131.92, 149.51, 206.81; MS (*m*/*z*): 423 (M^+).

*Compound (**4h**)*

mp: 200–202 °C (lit. 202–204 °C [24]); ^{1}H NMR (300 MHz, $CDCl_3$ δ ppm): 0.99 (12H, s, CH_3),2.46 (2H, s, CH_2), 2.70 (4H, d, J = 13.5 Hz, $COCH_2$), 2.81 (4H, d, J = 13.5 Hz, $COCH_2$), 3.61 (4H, s, NCH_2), 7.10 (2H, d, J = 8.5 Hz, ArH), 7.50 (2H, d, J = 8.5 Hz, ArH); ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 28.12, 28.58, 30.65, 32.24, 51.25, 54.83, 65.16, 113.38, 120.61, 131.33, 150.93, 205.26; MS (*m*/*z*): 487 (M^+).

Conclusion

In summary, we have developed an efficient synthesis of 3,5-dispirosubstituted piperidines of antibacterial profiles through domino sequence of Knoevenagel, Michael, and double Mannich reactions. The combination of highly stable and reusable iron(III) trifluroacetate Lewis acid with aq. SDS micellar medium has proven to be an effective catalytic system in contrast to other iron(III) metal salts for the synthesis of bis-spiro piperidines. This methodology is endowed with several advantages such as an inexpensive as well as nontoxic catalyst, easy work-up procedure, use of green micellar medium, and ambient temperature.

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