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A mild and efficient 1,4-addition of thiols and phenols to α,β-unsaturated carbonyl compounds using $La(NO_3)_3{\cdot}6H_2O$ as a catalyst under solvent-free conditions☆

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Abstract

A mild and efficient 1,4-addition of thiols and phenols to α,β-unsaturated carbonyl compounds in the presence of $La(NO_3)_3{\cdot}6H_2O$ under solvent-free conditions at room temperature in excellent yields is described.

Keywords: $La(NO_3)_3{\cdot}6H_2O$; 1,4-Addition; α,β-Unsaturated carbonyl compounds; Thiols; Phenols

1. Introduction

The 1,4-addition of thiols and phenols to α,β-unsaturated carbonyl compounds leading to the formation of C–S and C–O bond is very important transformation as they are constituents of key intermediates in synthesis of various natural products as well as in organic synthesis [1,2]. This has led to the development of novel synthetic methodologies for these compounds. Traditionally the addition to α,β-unsaturated carbonyl compounds has been reported with strong bases, such as alkali metal alkoxides, hydroxides and amines [3]. In recent years it has been noted that these reactions can also be promoted by Lewis acids, viz., $Zn(ClO_4)_2{\cdot}6H_2O$ [4], $Bi(OTf)_3$ [5], $InCl_3$ [6], $Cu(BF_4)_2$ [7], $Hf(OTf)_3$ [8] and $FeCl_3$ [9]. However, the use of either strongly acidic or basic conditions frequently leads to the formation of undesirable side products competing the reactions, such as polymerization, self-condensation and rearrangements, which in turn decrease the purity and yields of the desired products [10]. In view of current interest in catalytic processes, there is a merit in developing of 1,4-addition of thiols and phenols to α,β-unsaturated carbonyl compounds using inexpensive, mild and non-polluting reagent.

In the course of our on going search for chemoselective reagents, in multi-step synthesis of natural products, our group reported $La(NO_3)_3{\cdot}6H_2O$ as a mild and efficient catalyst for the chemoselective tetrahydropyranylation of primary alcohols [11], chemoselective deprotection of acetonides [12], synthesis of quinazolinones [13], mild and efficient acetylation of phenols and amines [14], α-amino nitriles [15], synthesis of benzodiazepines [16] and *N-tert*-butoxycarbonylation, *N*-benzyloxycarbonylation of amines [17,18]. $La(NO_3)_3{\cdot}6H_2O$ is a mild, inexpensive, comparatively non-toxic, readily available, easy to handle and insensitive to air. In the course of study in above transformations, it has been observed that the substrates containing other acid labile functional groups, such as TBDMS ethers, some isopropylidene protected diols and *N-tert*-Boc protected amines were intact in the presence of $La(NO_3)_3{\cdot}6H_2O$. In continuation of our work for the utility of $La(NO_3)_3{\cdot}6H_2O$, we found that it is an efficient and mild Lewis acid catalyst for 1,4-addition of thiols and phenols to α,β-unsaturated carbonyl compounds under solvent-free conditions.

2. Results and discussion

In this report (Scheme 1) we describe an efficient method for 1,4-addition of thiols and phenols to α,β-unsaturated carbonyl compounds in the presence of $La(NO_3)_3{\cdot}6H_2O$. This method does not need expensive reagents or special care to exclude the moisture from the reaction medium (solvent-free

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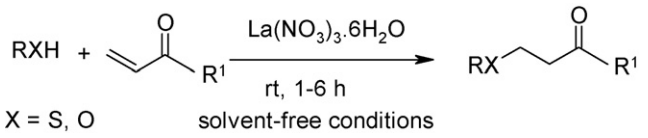
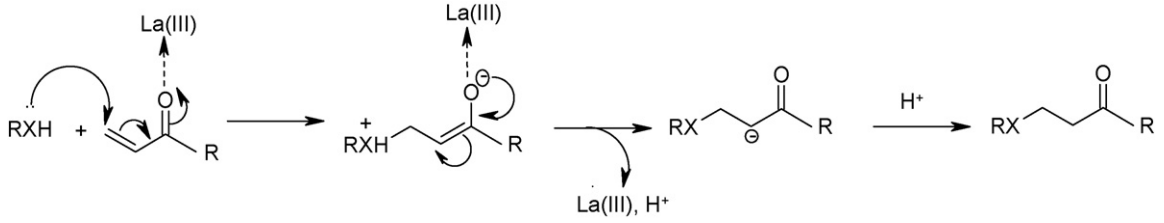


Scheme 1.

Table 1
Optimization of reaction conditions on the reaction of 2-thionapthol and methyl vinyl ketone using $La(NO_3)_3{\cdot}6H_2O$ as a catalyst

Entry	Solvent	$La(NO_3)_3{\cdot}6H_2O$ (mol%)	Time (h)	Yield (%)
1	DCM	100	12	50
2	THF	100	12	65
3	CH_3CN	100	12	75
4	$CHCl_3$	100	12	50
5	Dioxane	100	12	75
6	Neat	100	2	92
7	Neat	50	2	92
8	Neat	25	2	90
9	Neat	10	2	90
10	Neat	5	2	90

conditions). The reaction proceeded efficiently and smoothly at room temperature and the products are obtained in excellent yields. Furthermore, the reaction conditions are very mild, no by-products were observed. Lanthanum(III) nitrate hexahydrate is highly oxophilic and forms labile bond with carbonyl oxygen and initiate the formation of C–X bond with thiols/phenols (Scheme 2). This feature often allows substoichiometric amount of the catalyst to be used to promote a reaction. We first examined the reaction of methyl vinyl ketone (1 mmol) with 2-thionaphthol (1 mmol) in presence of $La(NO_3)_3{\cdot}6H_2O$ (5 mol%) at room temperature to give the corresponding keto-sulfide in 90% yield (Table 2, Entry 1). In order to optimize the reaction conditions, we carried out the above reaction in different solvents and also varied the amount of catalyst 5–100 mol%. Increasing the amount of $La(NO_3)_3{\cdot}6H_2O$ did not show significant influence on the rate of the reaction as well as yield (Table 1, Scheme 2). We found using 5 mol% of $La(NO_3)_3{\cdot}6H_2O$ under solvent-free conditions gave good to excellent yields (>90%) of the corresponding keto-sulfide. Whereas in the absence of the catalyst do not yield the corresponding product even after long reaction time (24 h). Encouraged by the success of this reaction using catalytic amount of $La(NO_3)_3{\cdot}6H_2O$, various thiols were reacted with differently substituted α,β-unsaturated carbonyl compounds to afford corresponding keto-sulfides in excellent yields (Table 2 , Scheme 1). Further we extended the veracity of the catalyst for addition of phenols to α,β-unsaturated carbonyl compounds, they are also efficiently converted into the corresponding keto-ethers (Table 3, Scheme 1). However, aliphatic alcohols do not yield the corresponding products with α,β-unsaturated carbonyl compounds (Table 3, Entries 11 and 12). From the foregoing results (Tables 2 and 3) it is evident that $La(NO_3)_3{\cdot}6H_2O$ is an



Scheme 2. Plausible mechanism.

Table 2
1,4-Addition of thiols to α,β-unsaturated carbonyl compounds catalyzed by $La(NO_3)_3{\cdot}6H_2O$ under solvent-free conditions

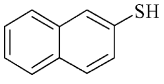
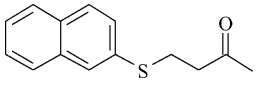
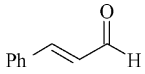
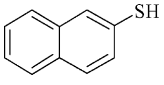
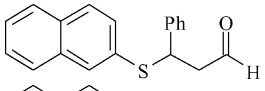
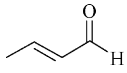
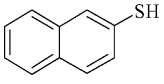
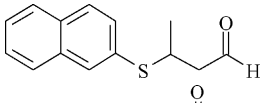
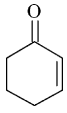
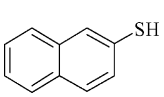
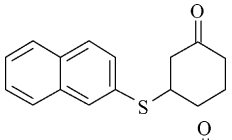
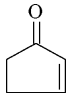
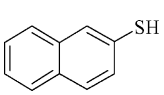
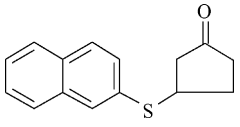
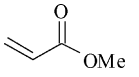
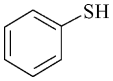
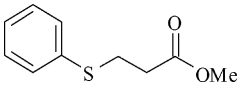
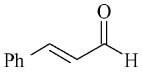
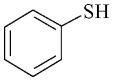
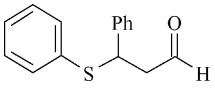
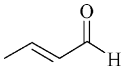
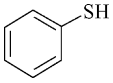
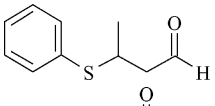
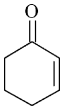
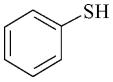
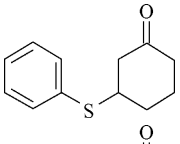
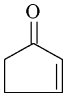
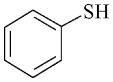
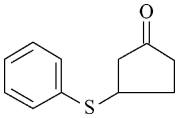
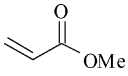
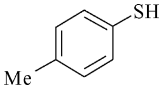
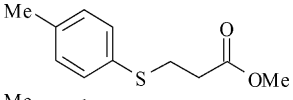
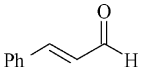
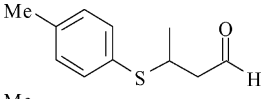
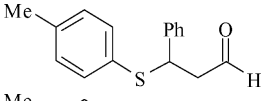
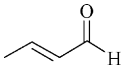
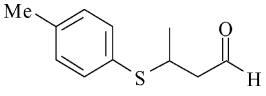
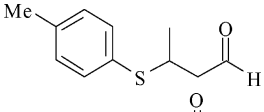
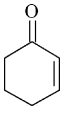
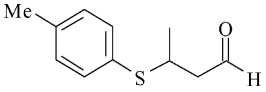
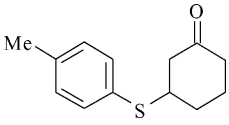
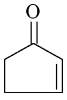
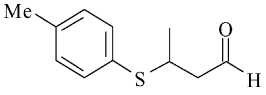
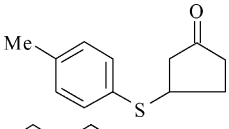
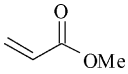
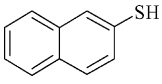
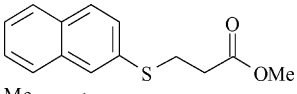
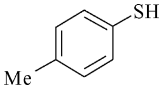
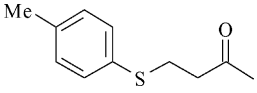
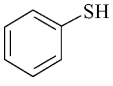
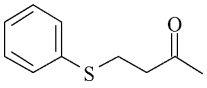
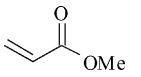
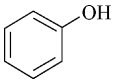
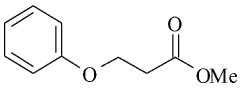
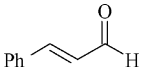
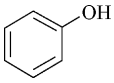
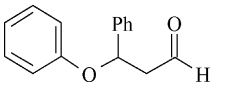
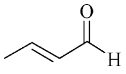
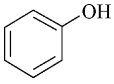
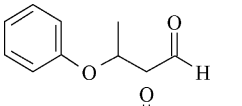
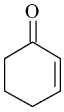
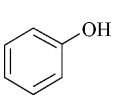
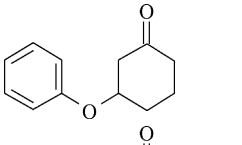
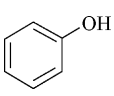
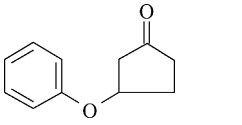
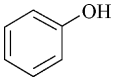
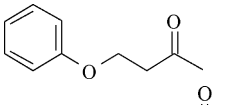
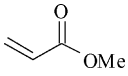
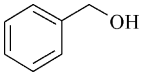
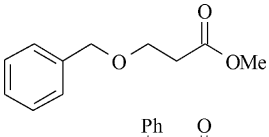
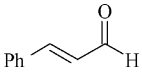
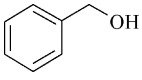
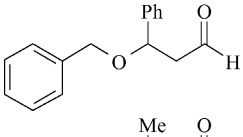
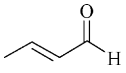
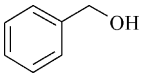
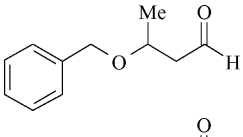
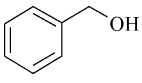
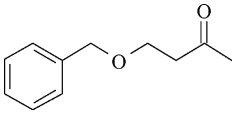
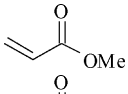
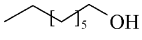
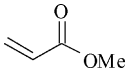
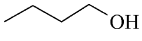
Entry	α,β-Unsaturated carbonyl compounds	Thiols	Product[a]	Time (h)	Yield (%)[b]
1				2	90
2				3	93
3				3	91
4				3	90
5				3	93

Table 2 (*continued*)

Entry	α,β-Unsaturated carbonyl compounds	Thiols	Product[a]	Time (h)	Yield (%)[b]
6				2	92
7				2	90
8				2	90
9				2	90
10				2	95
11				2	95
12				2	95
13				2	94
14				2	93
15				1	95
16				5	95
17				2	95
18				2	93
19		EtSH		2	90
20				1	95

[a] All the compounds were characterized by their spectral (^{1}H NMR and EIMS) data.
[b] Isolated yields after column chromatography.

Table 3
1,4-Addition of phenols to α,β-unsaturated carbonyl compounds catalyzed by $La(NO_3)_3{\cdot}6H_2O$ under solvent-free conditions

Entry	α,β-Unsaturated carbonyl compounds	Phenol	Product[a]	Time (h)	Yield (%)[b]
1				5	90
2				5	90
3				6	89
4				6	85
5				5	89
6				5	90
7				5	90
8				6	91
9				5	93
10				5	95
11			No reaction	5	–
12			No reaction	5	–

[a] All the compounds were characterized by their spectral (^{1}H NMR and EIMS) data.
[b] Isolated yields after column chromatography.

efficient catalyst for 1,4-addition of thiols and phenols to α,β-unsaturated carbonyl compounds under solvent-free conditions.

3. Conclusion

In conclusion, we described a mild and efficient method for the synthesis of keto-sulphides and keto-oxides by the reaction of thiols and phenols to α,β-unsaturated carbonyl compounds using $La(NO_3)_3{\cdot}6H_2O$ as a catalyst under solvent-free conditions. The method is having advantage of reduced reaction time, do not use of organic solvents, simple experimental and work-up procedure with high yields of products, which makes a useful addition to the present existing methodologies.

4. Experimental section

4.1. Typical experimental procedure

To a neat mixture of α,β-unsaturated carbonyl compounds (1 mmol) and thiol/phenol (1 mmol) was added $La(NO_3)_3{\cdot}6H_2O$ (5 mol%). The reaction was stirred at room temperature under solvent-free conditions for an appropriate time (Tables 2 and 3). After completion of the reaction as monitored by TLC, water (10 mL) was added to the reaction mixture and the product was extracted into ethyl acetate (3 × 20 mL). The combined organic layer was washed with brine, dried over anhydrous sodium sulphate and concentrated under reduced pressure to give crude

mass, which was purified over silica gel column chromatography to afford corresponding product in good yields.

Table 2, Entry 1. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.20$ (s, 3H, CH_3), 2.55 (t, 2H, CH_2), 3.15 (t, 2H, CH_2), 7.40 (m, 3H, Ar-H), 7.70 (m, 4H, Ar-H). EIMS: 230 ($M^{\bullet+}$).

Entry 2. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.90$ (m, 2H, CH_2), 4.52 (dd, 1H, CH, $J = 7.90$ Hz and 2.60 Hz), 7.39 (m, 3H, Ar-H), 7.41–7.45 (m, 5H, Ar-H), 7.75 (m, 4H, Ar-H), 9.50 (s, 1H, CHO). EIMS: 292 ($M^{\bullet+}$).

Entry 3. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.40$ (d, 3H, CH_3, $J = 6.90$ Hz), 2.62 (m, 2H, CH_2), 3.80 (m, 1H, CH), 7.41 (m, 3H, Ar-H), 7.72 (m, 4H, Ar-H), 9.81 (dd, 1H, CHO, $J = 6.50$ Hz and 3.50 Hz). EIMS: 230 ($M^{\bullet+}$).

Entry 4. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.65$ (m, 2H, CH_2), 2.21 (m, 4H, $2CH_2$), 2.5 (m, 2H, CH_2), 3.32 (m, 1H, CH), 7.41–7.45 (m, 3H, Ar-H), 7.70–7.74 (m, 4H, Ar-H). EIMS: 256 ($M^{\bullet+}$).

Entry 5. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.99$ (m, 2H, CH_2), 2.41 (m, 2H, CH_2), 2.50 (m, 2H, CH_2), 3.40 (m, 1H, CH), 7.30 (t, 1H, $J = 8.20$ Hz, Ar-H), 7.41 (m, 2H, Ar-H), 7.55 (s, 1H, Ar-H), 7.62 (d, 1H, $J = 8.52$ Hz), 7.80 (m, 2H, Ar-H). EIMS: 242 ($M^{\bullet+}$).

Entry 6. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.60$ (t, 2H, CH_2), 3.21 (t, 2H, CH_2), 3.70 (s, 3H, CH_3), 7.40 (m, 5H, Ar-H). EIMS: 196 ($M^{\bullet+}$).

Entry 7. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.91$ (m, 2H, CH_2), 4.50 (dd, 1H, CH), 7.10–7.60 (m, 10H, Ar-H), 9.45 (dd, 1H, CHO, $J = 6.80$ Hz and 2.20 Hz). EIMS: 242 ($M^{\bullet+}$).

Entry 8. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.33$ (d, 3H, CH_3), 2.60 (m, 2H, CH_2), 3.60 (m, 1H, CH), 7.21 (m, 5H, Ar-H), 9.70 (s, 1H, CHO). EIMS: 180 ($M^{\bullet+}$).

Entry 9. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.65$ (m, 2H, CH_2), 2.20 (m, 4H, $2CH_2$), 2.50–2.61 (m, 2H, CH_2), 3.30 (m, 1H, CH), 7.32 (s, 5H, Ar-H). EIMS: 206 ($M^{\bullet+}$).

Entry 10. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.90$ (m, 2H, CH_2), 2.21 (m, 2H, CH_2), 2.50 (m, 2H, CH_2), 3.38 (m, 1H, CH), 7.2–7.31 (m, 5H, Ar-H). EIMS: 192 ($M^{\bullet+}$).

Entry 11. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.30$ (s, 3H, Ar-CH_3), 2.60 (m, 2H, CH_2), 2.70 (m, 2H, CH_2), 3.60 (s, 3H, OCH_3), 7.20–7.31 (m, 5H, Ar-H). EIMS: 210 ($M^{\bullet+}$).

Entry 12. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.30$ (s, 3H, Ar-CH_3), 2.90 (m, 2H, CH_3), 4.50 (dd, 1H, CH), 7.20–7.60 (m, 9H, Ar-H), 9.45 (s, 1H, CHO). EIMS: 256 ($M^{\bullet+}$).

Entry 13. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.42$ (d, 3H, CH_3), 2.30 (s, 3H, Ar-CH_3), 2.70 (m, 2H, CH_2), 3.80 (m, 1H, CH), 7.20 (d, 2H, $J = 8.02$ Hz, Ar-H), 7.30 (d, 2H, $J = 8.02$ Hz, Ar-H), 9.50 (s, 1H, CHO). EIMS: 194 ($M^{\bullet+}$).

Entry 14. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.65$ (m, 2H, CH_2), 2.20 (m, 7H, $2CH_2$, Ar-CH_3), 2.50–2.61 (m, 2H, CH_2), 3.30 (m, 1H, CH), 7.30 (s, 4H, Ar-H). EIMS: 220 ($M^{\bullet+}$).

Entry 15. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.90$ (m, 2H, CH_2), 2.21 (m, 2H, CH_2), 2.25 (s, 3H, Ar-CH_3), 2.50 (m, 2H, CH_2), 3.38 (m, 1H, CH), 7.23–7.30 (m, 4H, Ar-H).

Entry 16. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.60$ (t, 2H, CH_2), 3.21 (t, 2H, CH_2), 3.70 (s, 3H, OCH_3), 7.40 (m, 3H, Ar-H), 7.70 (m, 4H, Ar-H). EIMS: 246 ($M^{\bullet+}$).

Entry 17. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.20$ (s, 3H, CH_3), 2.30 (s, 3H, Ar-H), 2.60 (t, 2H, CH_2), 3.00 (t, 2H, CH_2), 7.30 (m, 4H, Ar-H). EIMS: 194 ($M^{\bullet+}$).

Entry 18. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.20$ (s, 3H, CH_3), 2.60 (t, 2H, CH_2), 3.10 (t, 2H, CH_3), 7.10 (t, 1H, $J = 8.02$ Hz, Ar-H), 7.30 (t, 2H, $J = 8.02$, Ar-H), 7.40 (d, 2H, $J = 8.12$, Ar-H). EIMS: 180 ($M^{\bullet+}$).

Entry 19. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.10$ (s, 3H, CH_3), 2.15 (s, 3H, CH_3), 2.40 (m, 6H, $2CH_2$). EIMS: 132 ($M^{\bullet+}$).

Entry 20. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.15$ (s, 3H, CH_2), 2.50–2.60 (m, 4H, $2CH_2$), 3.74 (s, 2H, Ar-CH_2), 7.35–7.41 (m, 5H). EIMS: 194 ($M^{\bullet+}$).

Table 3. Entry 1. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.55$ (t, 2H, CH_2), 3.55 (s, 3H, OMe), 4.02 (t, 3H, CH_2), 6.50 (t, 1H, $J = 8.12$, Ar-H), 6.63 (d, 2H, $J = 8.15$, Ar-H), 7.24 (t, 2H, $J = 8.15$, Ar-H). EIMS: 180 ($M^{\bullet+}$).

Entry 2. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.95$ (m, 2H, CH_2), 5.45 (t, 1H, CH), 6.90 (m, 3H, Ar-H), 7.48 (m, 5H, Ar-H), 7.51 (m, 2H, Ar-H), 9.50 (s, 1H, CHO). EIMS: 226 ($M^{\bullet+}$).

Entry 3. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.48$ (d, 3H, CH_3), 2.70 (m, 2H, CH_2), 4.55 (dd, 1H, CH), 6.80 (m, 3H, Ar-H), 7.33 (d, 2H, Ar-H). EIMS: 164 ($M^{\bullet+}$).

Entry 4. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.95$–2.10 (m, 4H, $2CH_2$), 2.30–2.39 (m, 2H, CH_2), 2.50 (m, 2H), 6.05 (m, 1H, CH), 7.22 (m, 5H, Ar-H). EIMS: 190 ($M^{\bullet+}$).

Entry 5. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 1.50$ (m, 2H, CH_2), 2.23 (m, 4H, $2CH_2$), 4.70 (m, 1H, OCH), 7.33 (m, 5H, Ar-H). EIMS: 176 ($M^{\bullet+}$).

Entry 6. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.17$ (s, 3H, CH_3), 2.73 (t, 2H, CH_2), 4.07 (t, 2H, CH_2), 6.89–7.09 (m, 5H, Ar-H). EIMS: 164 ($M^{\bullet+}$).

Entry 7. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.62$ (t, 2H, CH_2), 3.76 (t, 2H, CH_2), 3.90 (s, 3H, OCH_3), 4.57 (s, 2H, Ar-CH_2), 7.30 (s, 5H, Ar-H). EIMS: 194 ($M^{\bullet+}$).

Entry 10. ^{1}H NMR ($CDCl_3$, 300 MHz), $\delta = 2.17$ (s, 3H, CH_3), 2.66 (t, 2H, CH_2), 3.75 (t, 2H, CH_2), 4.50 (s, 2H, Ar-CH_2), 7.32 (s, 5H, Ar-H). EIMS: 178 ($M^{\bullet+}$).

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