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Acid catalyzed Knoevenagel condensation of thiobarbituric acid and aldehyde at room temperature

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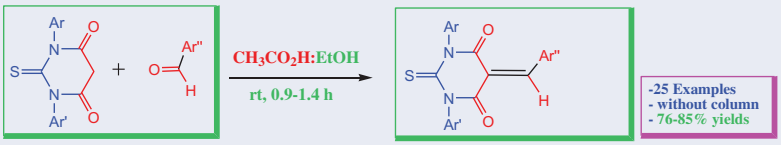
ABSTRACT

Knoevenagel reactions have been performed by the action of various unsymmetrical thiobarbituric acids containing activated methylene carbon and electron deficient center of aromatic aldehydes using small amount of acetic acid as initiator in ethanolic medium. The present protocol proceeded smoothly on room temperature stirring using ethyl alcohol as solvent with the help of initiator. The work-up procedure is very simple and products have been purified by simple recrystallization. Thus rendering the methodology is good and all synthesized molecules were characterized by ^{1}H, ^{13}C NMR, and MS spectra.

GRAPHICAL ABSTRACT



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KEYWORDS
Acetic acid catalyst; aldehydes; Knoevenagel products; TBAs

Introduction

The Knoevenagel reaction is a condensation between activated methylene carbon and carbonyl compounds in acidic or basic medium. The condensation is catalyzed by a weak base such as an amine and have powerful tool for carbon–carbon double bond formation strategy.[1] In the past few decades, several Knoevenagel condensation reactions have been promoted under distinct catalysts.[2,3] However few reports are most prominent on activated methylene carbon in water medium,[4] or with organocatalyst,[5] and In(III) catalyzed using acetic anhydride as promoter.[6] In fact, to increased electrophilicity or leaving-group ability of aldehydes in the presence of the acid additive could accelerate the Knoevenagel condensation which is further increased polar character due to carbon–carbon double bond network.

The Knoevenagel reaction is the most common synthetic strategy to produce electron deficient carbon–carbon double bond center. It has been widely employed in the preparation of benzylidene derivatives and important intermediates which is used in varied organic transformations. Therefore, alkylated and benzylidene derivatives of thiobarbituric acid have attracted attention of researchers toward medicinal chemistry,[7–11]

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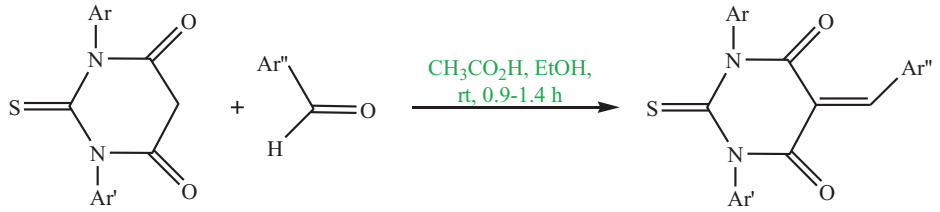
therapeutic drugs, pharmacological action[12–15] and some of them are more significant due to their diverse biological activity.[16] An insertion of an aryl, amino, or a methyl moiety at 5-position of thiobarbituric acid enhances the antidepressant activities[17] has been reported. Arylidene-pyrimidine-2,4,6-trione, arylidene-2-thioxo-dihydro-pyrimidine-4,6-dione and its derivatives were found to have hypotensive, transiquilizer and good anti-bacterial agents.[18,19] The barbituric acid and 2-thiobarbituric acid undergoes Knoevenagel condensations with aldehydes to give 5-substituted derivatives.[20,21] In addition, synthesis of benzylidenes, useful intermediate products, and also gives structural versatility in different nucleophiles, such as diketones, ketothioesters, 1,3-ketoesters[22,23] malonates, malononitriles[24] keto amides, and cyclic esters and with different aromatic[25] or aliphatic aldehydes.[26] Benzylidene thiobarbituric acids react with carbon nucleophiles[27,28] because of the fact that the active double bond in benzylidene carbon can easily be reduced[29,30] these compounds can be used for the synthesis of unsymmetrical disulfides[31,32] and for the mild oxidation of alcohols.[33,34] Kinetics of electrophilic alkylations of barbiturate and thiobarbiturate anions[35] and electrophilicity of 5-Benzylidene-1,3-dimethylbarbituric and -thiobarbituric acids[36] had been proved the strongly polarized exocyclic double bond.

TBAs have activated methylene carbon act as key precursor which is used in Knoevenagel condensation.[37] Recently we have reported solvent-free synthesis of Thiobarbituric acids as a precursor using Green Catalyst[38] and HPLC purification technique.[39] Effort to extend the continuation of our earlier work, here we wish to report the synthesis of benzylidene thiobarbituric acid derivatives by the reaction of thiobarbituric acid and aromatic aldehyde using catalytic amount of weak acid i.e., acetic acid in ethanolic medium at room temperature. The simple isolation and purification route used to make benzylidene derivatives is depicted in Scheme 1.

Results and discussion

We initially carried out the optimization by the reaction of 3-phenyl-2-thioxo-1-(4-methylphenyl)-dihydropyrimidine-4,6(1H,5H)-dione **1b** and 4-methoxybenzaldehyde 2 on room temperature stirring as model substrate and progress of the reaction has been monitored by TLC (Table 1). We examine the first reaction by loading 10 mol% of CH_3CO_2Na as a catalyst in solvent free condition, only 45% of **3b** was obtained at 4 h (entry 1). On the basis of first result, solvent may be important for the completion of reaction. Then we attempted the reaction with 10 mol% of CH_3CO_2Na in aqueous medium which offered 40% of **3b** (entry 2) because aromatic substituent is insoluble in water hence organic solvent is crucial for completion of reaction.

We planned to optimize the reaction condition by taking the mixture of CH_3CO_2Na:H_2O in 1:1, 1:2 and CH_3CO_2Na:H_2O:EtOH, 1:1:2 respectively, 48–58% of **3b** was obtained and **1b** remains unreacted (entries 3–5). Aqueous medium does not support completion of reaction hence by switching the aqueous medium to organic. Further optimization carried out by using mixture of 1:1 and 1:2 proportions of CH_3CO_2Na:EtOH, 66–73% of **3b** was obtained (entries 6 and 7). We have taken lot of effort to developing clean methodology using CH_3CO_2Na in varying medium but did not give notable impact.



1a-x **2** **3a-x**

3	1-Ar	3-Ar'	Ar"	3	1-Ar	3-Ar'	Ar"
a	Phenyl	Phenyl	4-Methoxyphenyl	**m**	4-Methoxyphenyl	Phenyl	4-Chlorophenyl
b	4-Methylphenyl	Phenyl	4-Methoxyphenyl	**n**	3-Chlorophenyl	Phenyl	4-Chlorophenyl
c	4-Methoxylphenyl	Phenyl	4-Methoxyphenyl	**o**	4-Methylphenyl	4-Methylphenyl	4-Chlorophenyl
d	2-Methylphenyl	2-Methylphenyl	4-Methoxyphenyl	**p**	3-Methylphenyl	4-Methylphenyl	4-Chlorophenyl
e	3-Methylphenyl	3-Methylphenyl	4-Methoxyphenyl	**q**	3-Chlorolphenyl	4-Methylphenyl	4-Chlorophenyl
f	4-Methylphenyl	4-Methylphenyl	4-Methoxyphenyl	**r**	3-Chlorolphenyl	4-Methoxyphenyl	4-Chlorophenyl
g	3-Methoxyphenyl	3-Methoxyphenyl	4-Methoxyphenyl	**s**	4-Chlorophenyl	4-Methoxyphenyl	4-Chlorophenyl
h	4-Methoxylphenyl	4-Methoxylphenyl	4-Methoxyphenyl	**t**	4-Chlorophenyl	Ethyl	4-Chlorophenyl
i	3-Chlorophenyl	3-Chlorophenyl	4-Methoxyphenyl	**u**	4-Methoxyphenyl	4-Methyl phenyl	4-Chlorophenyl
j	4-Chlorophenyl	4-Chlorophenyl	4-Methoxyphenyl	**v**	4-Chlorophenyl	4-Methylphenyl	4-Chlorophenyl
k	Phenyl	Phenyl	4-Chlorophenyl	**w**	3-Methoxyphenyl	4-Methylphenyl	4-Chlorophenyl
l	4-Methylphenyl	Phenyl	4-Chlorophenyl	**x**	4-Methylphenyl	Ethyl	4-Chlorophenyl

Scheme 1. The reaction is given above.

No significant improvement was observed in the present optimization, therefore, we changed the strategy by loading the catalytic amount of weak acid i.e., acetic acid to afford 49% of **3b** in absence of solvent (entry 8). When the reactions were carried out in different proportions of CH_3CO_2H:H_2O & CH_3CO_2H:H_2O:EtOH) did not give appreciable results (entries 9–12).

Therefore we confirmed that the aromatic substituent present in **1b** is more easily soluble in organic solvent hence, ethanol medium is good option to carried out the further reaction protocol. It was interesting to expand the scope for completion of reaction we carried out the same reaction by catalytic amount of 1:1 proportion of CH_3CO_2H:EtOH, 76% and at 1:2 proportion, 84% of **3b** were isolated (entries 13–14).

Encouraged this result (entry 14), we compared the ability by reducing the mol % of CH_3CO_2H & EtOH, low to moderate yields were obtained (entries 15–17). In all the cases studied, CH_3CO_2H:EtOH (1:2) was found to be the best choice for the rest reactions to give corresponding 5-arylbenzylidene-1,3-diaryl-2-thioxodihydropyrimidine-4,6-(1H,5H)-diones (3a-x) in excellent yields. The results are summarized in Table 2.

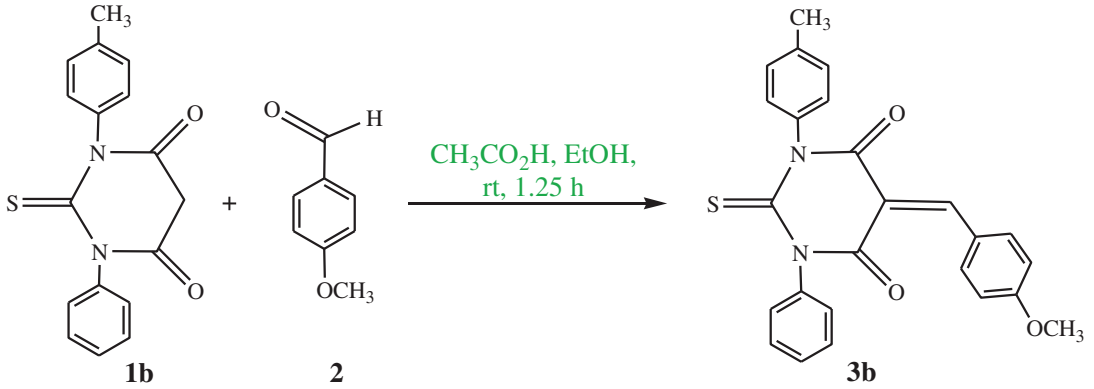
Material and methods

Experimental method

Synthesis of 5-(4-methoxybenzylidene)-1-(4-methylphenyl)-3-phenyl-2-thioxodihydro-pyri-midine-4,6-(1H,5H)-dione (3b)

5-(4-Methoxybenzylidene)-1-(4-methylphenyl)-3-phenyl-2-thioxodihydropyrimidine-4,6-(1H,5H)-dione (**3b**) was synthesized by the interaction of 1-(4-methylphenyl)-3-phenyl-

Table 1. Optimization of reaction conditions of 1-(4-methylphenyl)-3-phenyl-2-thioxo-dihydropyrimidine-4,6(1H,5H)-dione (**1b**) and 4-methoxybenzaldehyde[d] (**2**).



1b **2** **3b**

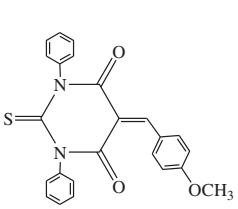
Entry	Catalyst	Loading (mol%)	Time (h)	Yield[e] (%)
1	CH_3CO_2Na	10	4.0	35[f]
2	$[CH_3CO_2Na : H_2O]$[a]	10	3.5	30[f]
3	$[CH_3CO_2Na : H_2O : EtOH]$[a]	10	3.5	40[f]
4	$[CH_3CO_2Na : H_2O : EtOH]$[b]	10	3.5	42[f]
5	$[CH_3CO_2Na : H_2O : EtOH]$[c]	10	3.5	50[f]
6	$[CH_3CO_2Na : EtOH]$[a]	10	3.5	58
7	$[CH_3CO_2Na : EtOH]$[b]	10	3.5	65
8	CH_3CO_2H	10	3.5	43[f]
9	$[CH_3CO_2H : H_2O]$[a]	10	3.5	40[f]
10	$[CH_3CO_2H : H_2O : EtOH]$[a]	10	3.5	46[f]
11	$[CH_3CO_2H : H_2O : EtOH]$[b]	10	3.5	49[f]
12	$[CH_3CO_2H : H_2O : EtOH]$[c]	10	3.5	51[f]
13	$[CH_3CO_2H : EtOH]$[a]	10	3.5	65
14	**$[CH_3CO_2H : EtOH]$[b]**	**10**	**1.25**	**84**
15	$[CH_3CO_2H : EtOH]$[b]	7.5	3.5	63
16	$[CH_3CO_2H : EtOH]$[b]	5.0	3.5	55
17	$[CH_3CO_2H : EtOH]$[b]	2.5	3.5	40[f]

Mole proportion of catalyst and solvent (1:1)[a]; (1:2)[b];(1:1:2)[c]; General reaction conditions[d]: 1-(4-methylphenyl)-3-phenyl-2-thioxo-dihydropyrimidine-4,6(1H,5H)-dione (10 mmol), 4-methoxybenzaldehyde (10 mmol); [e]present yield, [f]1b not consumed completely.

Optimized reaction condition has been found as significant, hence rest of the products have been prepared using same condition.

2-thioxo-dihydropyrimidine-4,6(1H,5H)-dione, (**1b**, 310 mg, 1 mmol) and p-methoxy benzaldehyde (2, 136 mg, 1 mmol) was loaded with 10 mol % of acetic acid (0.57 mL) and ethanol (1.04 mL) in 1:2 proportion at room temperature stirring over 1 h. The progress of reaction was monitored by TLC. After completion of reaction, mixture was dissolved in ethyl acetate then washed with brine solution followed by water. Solvent was evaporated under vacuum; the obtained residue was dried and finally purified by recrystallization from ethanol (305 mg, 84%). Yellow solid, mp 261–263 °C. ^{1}H NMR 400 MHz, ($CDCl_3$) ppm: δ 8.63 (s, 1H, =C–H), 8.44–8.41 (m, 2H $C_{2,}C_6$-Ar″), 7.52–7.50 (m, 2H, $C_{2,}C_{6,}$-Ar), 7.47–7.45 (m, 3H, $C_{3,}C_{4,}C_5$-Ar), 7.33–7.26 (m, 2H, $C_{2,}C_6$-Ar′), 7.19–7.15 (m, 2H, $C_{3,}C_5$-Ar′), 6.95–6.93 (m, 2H, $C_{3,}C_5$-Ar″), 3.91 (s, 3H, Ar″OCH_3), 2.42 (s, 3H, Ar′CH_3). 13C NMR (100 MHz, $CDCl_3$) ppm: δ 21.60, 55.93, 114.49, 114.76, 125.99, 128.50, 128.81, 128.95, 129.66, 130.49, 137.40, 138.94, 139.60, 140.20, 161.46, 162.51, 165.43, 180.81. HRMS (*m/z*) 429.1272 $[M+H]^+$, Calcd. 429.1267 $[M+H]^+$.

Table 2. Structure of different functionalized products.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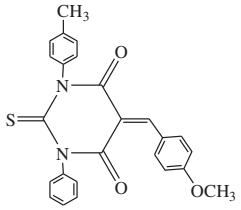
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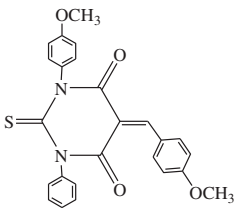
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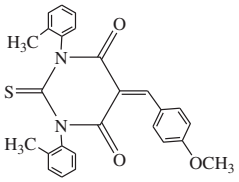
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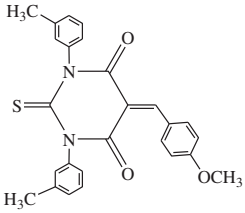
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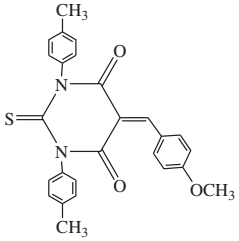
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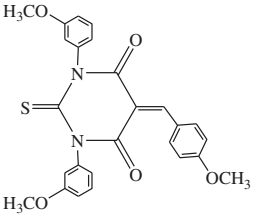
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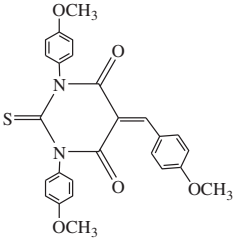
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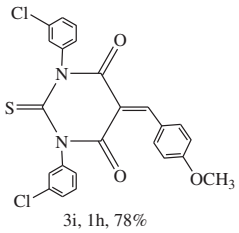
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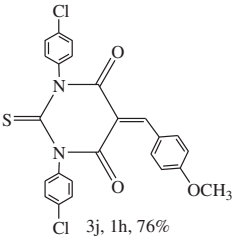
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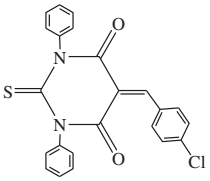
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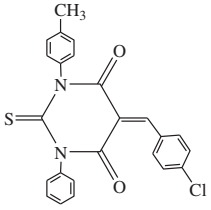
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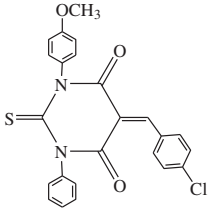
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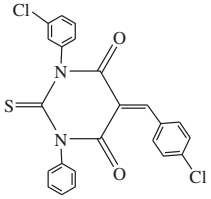
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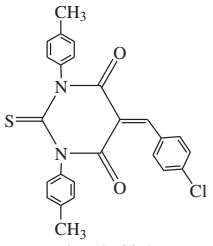
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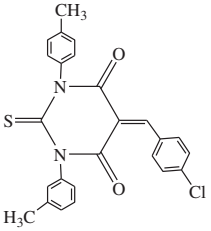
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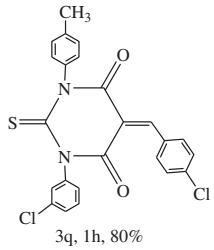
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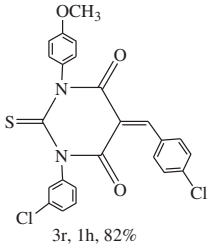
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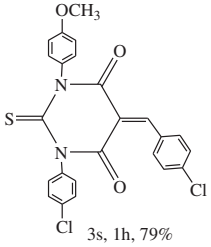
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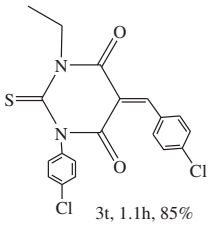
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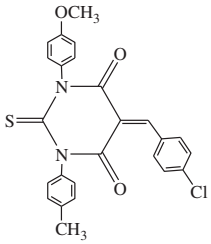
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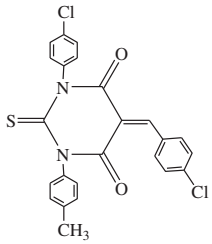
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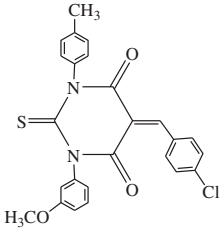
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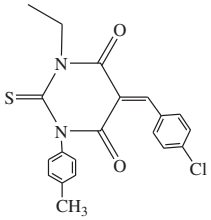
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Conclusion

We have developed a simple efficient approach for the synthesis of a series of 5-substituted arylbenzylidene-1,3-diaryl-2-thioxodihydropyrimidine-4,6-(1H,5H)-diones under simple room temperature stirring. This present protocol is considered as eco-friendly, inexpensive, offer the advantage of easier operational simplicity, simple reaction workup and purified by crystallization without column chromatography. All synthesized products (3a-x) were characterized and confirmed by ^{1}H, 13C NMR, HRMS and MS techniques.

^{1}H, 13C NMR and MS spectra of all synthesized compounds is provided in supplementary file.

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