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FULL PAPER

Salicylic Acid-catalyzed Three-component Synthesis of 1-Amido/thioamidoalkyl-2-naphthols Under Solvent-free Conditions

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Abstract:

A simple, efficient, and green procedure for the solvent-free synthesis of 1-amido/thioamidoalkyl-2-naphthols by one-pot stirring of starting reactants at 100 °C has been introduced. By utilizing salicylic acid (SA) as the catalyst, the reaction of aryl aldehydes, 2-naphthol, and acetamide/benzamide/urea/thiourea were successfully performed, and 1-amido/thioamidoalkyl-2-naphthols were obtained in good to high isolated yields. The notable advantages of this method over some previous methods are the availability of catalyst, atom efficiency, not using any solvent for reaction, relatively green conditions, no need for catalyst synthesis, relatively shorter reaction times, and no specialized equipment (microwave or ultrasound) or purification techniques are needed.

Keywords: amidoalkylnaphthols; *β*-naphthol; three-component reaction; salicylic acid; solvent-free

1. Introduction

The multi-component reactions (MCRs) have been recognized as a class of chemical transformations, which are useable for the construction of various synthetically useful heterocyclic compounds, natural products, complex libraries of biological molecules, and other organic molecules such as 1-amido/thioamidoalkyl-2-naphthols and different functionalized organic compounds in atom-economical ways by generating multiple bonds in a single step. MCRs can reduce the number of steps and present advantages including, low energy consumption, simplicity, ease of processing for isolation and purification, high efficiency, the lower making of chemical byproducts, little to no waste production, bond-forming economy, and leading to desired environmentally friendly processes [1-7]. MCRs under solvent-free reaction conditions (SFRCs) are also interesting since they involve the best reaction medium with “no medium” [8].

1-Amidoalkyl-2-naphthol derivatives are a class of organic compounds, which act as important building blocks for the synthesis of biologically important natural products, various drug-like molecules, such as aminoalkyl naphthols [9] and 1,3-oxazine frameworks [10] having hypotensive, bradycardiac, HIV protease inhibitory, anti-Parkinson’s, and antibacterial activities [11-14]. A one-pot, three-component Mannich type reaction between 2-naphthol, aldehydes, and amides has been used as a practical synthetic route toward the 1-amidoalkyl-2-naphthols [15]. Some new methods have been developed to improve the reaction yield in the synthesis of 1-amidoalkyl-2-naphthols using a wide variety of catalysts, including zirconocene dichloride (Cp_2ZrCl_2) [16], magnetite (Fe_3O_4)-supported molybdenum oxide (MoO_3) [17], magnetic nanoparticle-supported ionic liquid [18], graphene oxide [19], tannic acid [11], nano silica sulfuric acid [20], $ZrOCl_2 \cdot 8H_2O$ [21], nano-ZnO [22], lignosulfonic acid (LSA) [23], Fe_3O_4-nanoparticles grafted 1-methyl-3-(3-trimethoxysilylpropyl) imidazolium acetate (Fe_3O_4@IL-OAc) under sonication [24], phosphoric acid supported on alumina (H_3PO_4/Al_2O_3) [25], sulfonated polynaphthalene

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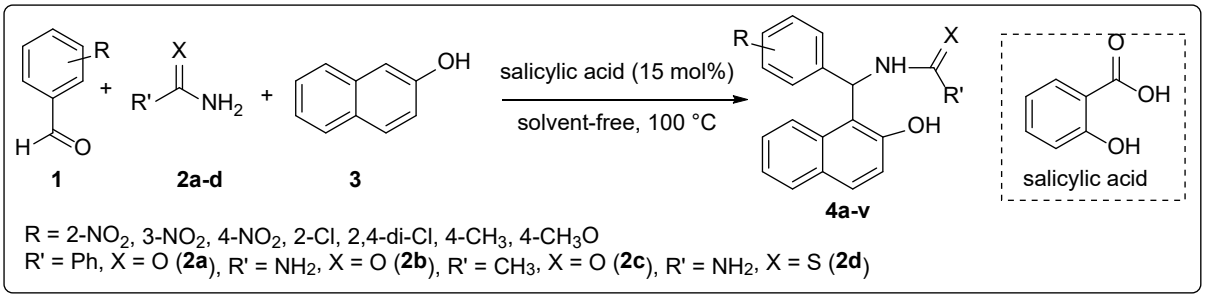
(S-PNP) [26], lemon juice [27], *β*-cyclodextrin-butane sulfonic acid [28], activated Fuller's earth [29], *β*-cyclodextrin-monosulphonic acid [30], 1-methylimidazolium tricyanomethanide {[HMIM]C(CN)$_3$} and SnO_2 nanoparticles [31], sulfanilic acid [32], magnetic nanoparticle supported acidic ionic liquid (AIL@MNP) [33], silver nanoparticles [34], and antimony (III) acetate [35]. Moreover, Szatmári and coworker are also reviewed other catalysts for similar transformation [36].

Salicylic acid (2-hydroxybenzoic acid) is a plant phenolic acid, which found in willow bark, willow leaves, poplar tree and has been used as an analgesic 400 B.C. Salicylic acid (SA) shows anti-bacterial, antifungal, antipyretic, and anti-inflammatory properties and also participates in many important activities in plants, including flowering, seed germination, heat production, membrane permeability, defense responses, and response to stress. This natural organoacid is employed as a precursor to the synthesis of aspirin®. Moreover, SA and its derivatives exist in skin-care products and are used for the treatment of acne [37-46].

The SA was found to catalyze some organic reactions like oxidation of alkenes [47], the Hantzsch multi-component reaction [48], hydrodeamination of aromatic amines [49], Sonogashira-type cross-coupling [50], and ring-opening polymerization (ROP) of ε-caprolactone [51]. Moreover, SA was used as a simple organocatalyst for the synthesis of diarylmethanones [52], *α*-aryl ketones [53], pyrrolidine derivatives [54], 2,3-dihydroquinazolin-4(1*H*)-ones [55], homoallylic alcohols [56] as well as 3,3′-bis(indolyl)methanes (BIMs) [57].

Due to a variety of applications of the described amidoalkyl naphthol compounds, and in continuation of our previous works on the synthesis of amido/thioamidoalkyl naphthols [58, 59], in this contribution, the catalytic activity of SA as an efficient and cost-effective organocatalyst has been explored for the synthesis of 1-amido/thioamidoalkyl-2-naphthols (**4a-v**) in MCR under SFRCs protocol (Scheme 1).



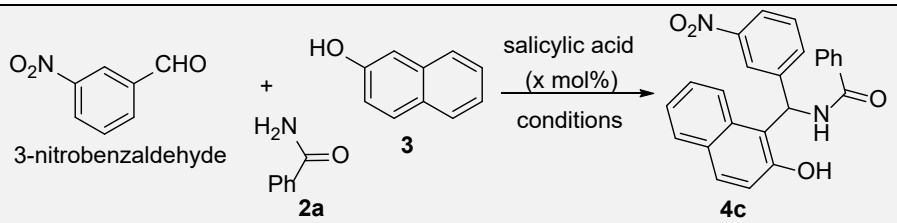
Scheme 1. Synthesis of 1-amido/thioamidoalkyl-2-naphthols (**4a-v**) in MCR under SFRCs at 100 ºC.

2. Results and Discussion

Initial experiments were performed using 3-nitrobenzaldehyde (1 mmol), benzamide (**2a**, 1 mmol), and 2-naphthol (**3**, 1 mmol) as the model reaction. The results are summarized in Table 1. It is disappointing that the reaction gave no desired product (**4c**) in the absence of a catalyst at refluxing water after 120 min (Table1, entry 1). When the reaction was performed in the presence of SA (5 mol%) at refluxing water, the corresponding product (**4c**) was obtained in 25% isolated yield (Table 1, entry 2). No better yield was achieved when the reaction was carried out in water in the presence of various amounts of catalyst (Table 1, entries 3 and 4). Implementation of the reaction in other solvents such as ethanol, acetonitrile, and dichloromethane did not achieve good results (Table 1, entries 5-7). The reaction was performed in solvent-free conditions (SFRCs) at 100 °C without any catalyst, and the desired product **4c** was observed in minor amounts as well as even after prolonged reaction time a significant amount of starting materials was intact (Table 1, entry 8). When the 5 mol% SA catalyst was added to the reaction mixture at 100 °C under SFRC, the product was obtained with 83% isolated yield after 50 min (Table 1, entry 9). The yield was increased to 85% and 92% when 10

and 15 mol% of SA was employed, respectively (Table 1, entries 10 and 11). There was no increase in the reaction yield when 20 mol% of catalyst was used (Table 1, entry 12). Screening various temperatures did not get satisfactory results (Table 1, entries 13-16). With these interpretations, we conclude that 15 mol% of the catalyst, the temperature of 100 °C and the SFRCs, are the best conditions for the reaction.

Table 1. Optimization of the reaction conditions.[a]



Entry	Solvent	Temp. (°C)	SA (mol%)	Time (min)	Isolated yields (%)
1	H_2O	reflux	-	120	trace
2	H_2O	reflux	5	120	25
3	H_2O	reflux	10	120	45
4	H_2O	reflux	15	120	55
5	EtOH	reflux	15	120	47
6	CH_3CN	reflux	15	120	52
7	CH_2Cl_2	reflux	15	120	28
8	SF[b]	100	-	120	trace
9	SF	100	5	50	83
10	SF	100	10	40	85
11[c]	**SF**	**100**	**15**	**30**	**92**
12	SF	100	20	30	93
13	SF	50	15	120	45
14	SF	60	15	120	77
15	SF	80	15	55	80
16	SF	120	15	38	86

[a] Reaction conditions: the reaction was performed using a mixture of 3-nitrobenzaldehyde (1 mmol), benzamide (**2a**, 1 mmol), 2-naphthol (**3**, 1 mmol), and catalyst (x mol%) under SFRCs or solution state. [b] Solvent free. [c] Optimized conditions.

In the direction of search the scope of this 3-CR, some substituted benzaldehydes reacted with amides (benzamide, urea, acetamide)/thiourea (**2a-d**) and 2-naphthol (**3**) under the optimized reaction conditions, and the results are shown in Table 2. All the corresponding 1-amido/thioamidoalkyl-2-naphthols (**4a-v**) were synthesized in good to excellent isolated yields without any special purification methods, although in some cases the reactions proceeded rather slowly.

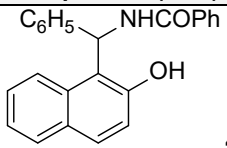
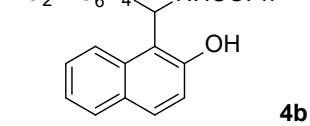
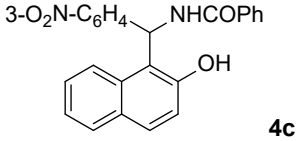
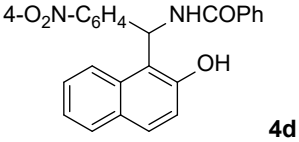
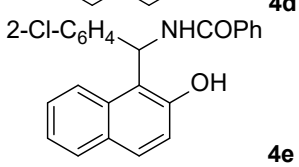
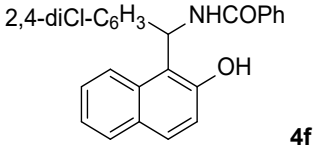
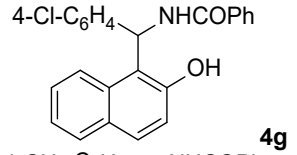
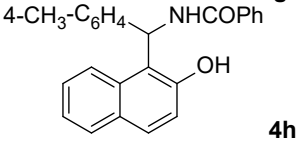
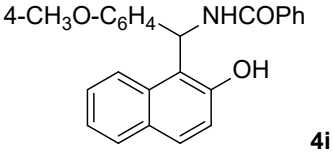
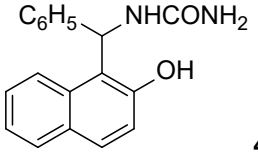
Electron-donating, electron-withdrawing containing substituted benzaldehydes and sterically hindered those such as 2-nitrobenzaldehyde, 2-chlorobenzaldehyde, and 2,4-dichlorobanzaldehyde are well reacted in this 3-CR to afford the desired products.

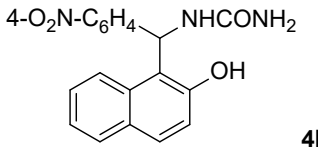
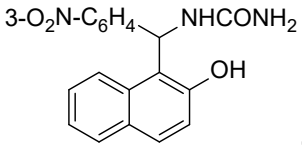
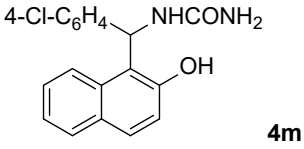
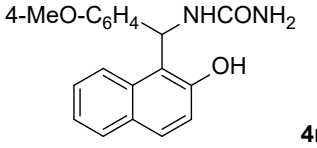
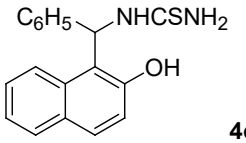
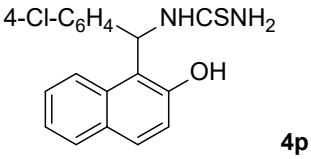
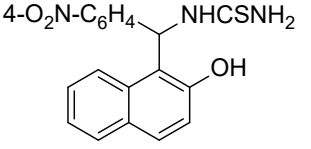
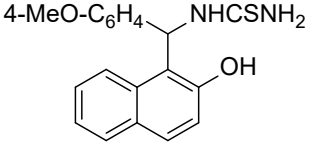
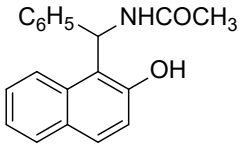
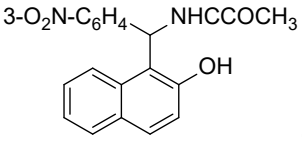
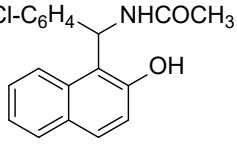
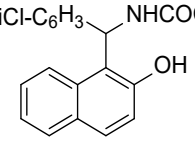
To display the efficiency of the SA as a commercially cost-effective organocatalyst with other catalysts, several results for the synthesis of *N*-((2-hydroxynaphthalen-1-yl)(4-nitrophenyl)methyl)benzamide (**4d)** from 4-nitrobenzaldehyde, benzamide (**2a**), and 2-naphthol (**3**) are presented in Table 3. SA Clearly showed catalytic activity regarding reaction times and product yields. Moreover, current work does not require synthesis of catalyst [16, 18, 28, 30, 31], hazardous solvents such as ethylene dichloride (EDC) [16] and dichloroethane (DCE) [18] or no need for special devices such as ultrasound [18].

The possible reaction mechanism for the formation of 1-amido/thioamidoalkyl-2-naphthols (**4a-v**) is presented in Scheme 2. Probably the reaction starts through the activation of substituted benzaldehydes by the SA catalyst. In

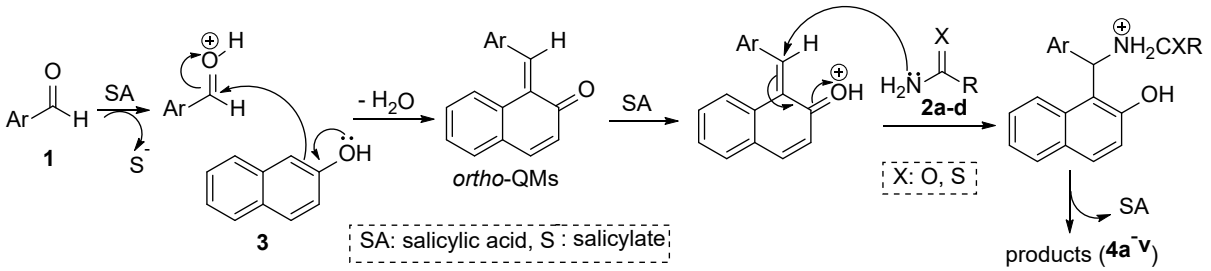
the second step, the *ortho*-quinone methides (*o*-QMs) were formed *via* the reaction of activated aldehydes and 2-naphthol. The Michael addition of compounds **2** into the *o*-QMs intermediates, and then elimination of water leads to the creation of desired products (**4a-v**).

Table 2. The three-component synthesis of substituted 1-amido/thioamidoalkyl-2-naphthols (**4a-v**) under SFRCs.[a]

Entry	Structure of 1-amido/thioamidoalkyl-2-naphthols (4a-v)	Time (min)	Isolated yields (%)	Mp (°C)	
				Found	Reported [ref.]
1	 **4a**	28	89	229-231	230-231 [59]
2	 **4b**	19	75	266-269	260-263 [62]
3	 **4c**	30	92	233-235	242-243 [33]
4	 **4d**	20	93	237-238	239-241 [62]
5	 **4e**	20	97	266-268	266-268 [62]
6	 **4f**	12	95	234-236	235-238 [62]
7	 **4g**	12	93	177-178	179 [16]
8	 **4h**	35	85	210-213	211-213 [62]
9	 **4i**	34	88	206-208	207-209 [62]
10	 **4j**	22	90	178-181	178-182 [59]

11	 **4k**	18	94	192-194	190-193 [59]
12	 **4l**	24	94	192-194	197-198 [62] 190-192 [59]
13	 **4m**	18	92	170-172	168-169 [60] 168-170 [59]
14	 **4n**	30	88	185-187	186-187 [60]
15	 **4o**	25	89	180-181	179-181 [60]
16	 **4p**	23	93	176-178	175-177 [60]
17	 **4q**	20	93	178-179	176-178 [60]
18	 **4r**	32	86	138-140	136-138 [60]
19	 **4s**	25	90	241-243	241-244 [59]
20	 **4t**	20	91	266-268	265-269 [59]
21	 **4u**	10	97	210-211	208-209 [62]
22	 **4v**	15	97	230-231	201-203 [33]

[a] Reaction conditions: substrates (1 mmol); SA (15 mol%); heating at 100 ºC.



Scheme 2. The proposed mechanism for the synthesis of 1-amido/thioamidoalkyl-2-naphthols (**4a-v**) using SA catalyst.

Table 3. Comparison of the synthesis of *N*-((2-hydroxynaphthalen-1-yl)(4-nitrophenyl)methyl)benzamide (**4d)** using SA with other reported catalysts.

Entry	Catalyst	Conditions	Time (min)	Yield (%)	References
1	Cp_2ZrCl_2	EDC, rt	360	90	[16]
2	IL@MNP	DCE, US, 30 ºC	18	94	[18]
3	IL@MNP	DCE, stirring, 30 ºC	50	56	[18]
4	$ZrOCl_2.8H_2O$	SF, 80 °C	40	88	[21]
5	*β*-CD-BSA	SF, 100 °C	10	94	[28]
6	Activated Fuller's earth	SF /110 °C	12	93	[29]
7	*β*-CD-mono SO_3H	SF, 80 °C	12	92	[30]
8	{[HMIM]C$(CN)_3$}	SF, rt	8	96	[31]
9	SnO_2 nanoparticles	SF, rt	20	87	[31]
10	Zinc benzenesulfonate	SF, 80 °C	60	89	[62]
11	SA	SF, 100 °C	20	93	This work

Cp_2ZrCl_2 Zirconocene dichloride; *EDC* ethylene dichloride; *IL@MNP* magnetic nanoparticle-immobilized ionic liquid; *DCE* dichloroethane; *US* ultrasonic; *CD* cyclodextrin; *SF* Solvent-free; *β*-CD-BSA *β*-cyclodextrin-butane sulfonic acid; {[HMIM]C$(CN)_3$}1-methylimidazolium tricyanomethanide; *rt* room temperature.

3. Material and Methods

All chemicals were purchased from Alfa Aesar and Aldrich as well as were used without further purification, except 4-methylbenzaldehyde, 4-methoxylbenzaldehyde, and benzaldehyde which were distilled before use. All solvents were distilled before use. The products were characterized by comparison of their physical data with those of known samples or by their spectral data. Melting points were measured on a Buchi 510 melting point apparatus. NMR spectra were recorded at ambient temperature on a BRUKER AVANCE DRX-400 MHz using DMSO-d_6 as the solvent. FT-IR spectra were recorded on a Perkin-Elmer RXI spectrometer. The development of reactions was monitored by thin layer chromatography (TLC) analysis on Merck pre-coated silica gel 60 F_{254} aluminum sheets, visualized by UV light. Elemental microanalyses were performed on an Elementar Vario EL III analyzer. All of the targeted products are reported in the literature, and they are shown to be identical with the authentic samples prepared according to our previous works [58, 59].

General procedure for the synthesis of 1-amido/thioamidoalkyl-2-naphthols (4a-v): A mixture of substituted aldehyde **1** (1 mmol), **2a-d** (benzamide, **2a**; urea, **2b**; acetamide, **2c**; thiourea, **2d**) or 2-naphthol **3** (1 mmol), and salicylic acid (15 mol%) was stirred at 100 ºC in an oil bath. After completion of the reaction (using TLC analysis), the reaction mixture was allowed to cool to room temperature. Next, hot ethyl acetate was added to the resulting mixture and then cooled to room temperature (rt). The resulting solid product was filtered off, washed with distilled water, and dried to afford the targeted compounds. If further purification was needed, the target compounds could be recrystallized from hot ethanol. Spectral data for compounds **4c** and **4t** were as follows:

*N-((2-Hydroxynaphthalen-1-yl)(3-nitrophenyl)methyl)benzamide (**4c**)*: IR (KBr): 3369 (N-H amide, str), 3263 (O-H, Ar, str), 3093 (C-H, Ar, str), 1633 (C=O amide, str), 1531,1346 (N-O_2, str), 1439 (C=C, Ar, str), 812 (C-H, Ar, out of plane, bend), 740-653 (N-H, out of plane, bend); ^{1}H NMR (400 MHz, DMSO-d_6): δ = 10.48 (s, 1H, OH), 9.19 (d, *J* = 7.9 Hz, 1H, Ar-H), 8.16

(s, 1H, Ar-H), 8.13 (d, *J* = 8.5 Hz, 1H, Ar-H), 8.09 (d, *J* = 8.01 Hz, 1H, Ar-H), 7.92 (d, *J* = 7.7 Hz, 2H, Ar-H), 7.86-7.84 (m, 2H, NH, Ar-H), 7.74 (d, *J* = 7.7 Hz, 1H, Ar-H), 7.59-7.54 (m, 2H, Ar-H), 7.50-7.44 (m, 4H, Ar-H), 7.33 (d, *J* = 7.4 Hz, 1H, Ar-H), 7.29 (d, *J* = 9.1 Hz, 1H, Ar-H); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 167.2 (C=O), 154.4 ($C_{ar\text{-}OH}$), 148.7, 145.4, 134.9, 134.2,133.1, 132.4, 130.9, 130.6, 129.6, 129.3, 129.3, 128.3, 127.9, 123.7, 123.4, 122.5, 121.9, 119.5, 118.2, 49.8 (CH-NH).

N-((2-Hydroxynaphthalen-1-yl)(3-nitrophenyl)methyl)acetamide (***4t***): IR (KBr): 3374 (N-H amide, str), 3197 (O-H, Ar, str), 3089 (C-H, Ar, str), 1646 (C=O amide, str), 1523,1349 (NO_2, str), 1437 (C=C, Ar, str), 805 (C-H, Ar, out of plane, bend), 713-740 (N-H, out of plane, bend); ^{1}H NMR (400 MHz, DMSO-d_6): δ = 10.17 (s, 1H, OH), 8.65 (d, *J* = 8.0 Hz, 1H, Ar-H), 8.55 (d, *J* = 7.6 Hz, 1H, Ar-H), 8.02 (s, 1H, H_a), 7.86-7.81 (m, 3H, NH, Ar-H), 7.59-7.52 (m, 2H, Ar-H), 7.42 (t, *J* = 7.6 Hz, 1H, Ar-H), 7.29 (t, *J* = 7.5 Hz, 1H, Ar-H), 7.23 (d, *J* = 8.8 Hz, 1H, Ar-H), 7.19 (d, *J* = 7.9 Hz, 1H, Ar-H), 2.03 (s, 3H, CH_3); ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.6 (C=O), 154.2 ($C_{ar\text{-}OH}$), 148.6, 146.3, 133.7, 133.0, 130.8, 130.5, 129.6, 129.3, 127.5, 123.5, 122.1, 121.3, 119.3, 118.6, 48.4 (CH-NH), 23.4 (CH_3).

4. Conclusions

In conclusion, an efficient one-pot, three-component reaction of aromatic aldehydes, 2-naphthol, and amides/urea/thiourea aimed at the synthesis of amido/thioamidoalkyl naphthols under SFRCs has been developed. Easy workup, simplicity, cost-effective, easy purification, ready availability of the catalyst, and no need for special equipment, such as ultrasonic devices are remarkable benefits of this synthetic method

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