One-pot three-component synthesis of 1-amidoalkyl-2-naphthols in the presence of phthalimide-*N*-sulfonic acid

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A simple one-pot three-component reaction (3CR) was carried out using phthalimide-*N*-sulfonic acid (PISA) as a solid acid organocatalyst for synthesis of a variety of 1-amidoalkyl-2-naphthols. The reaction was conducted in order to preparation of 1-amidoalkyl-2-naphthols under solvent-free reaction conditions at 100 °C within 4-18 minutes and the corresponding products were formed in 75-97 % yields. The catalyst can be recovered and reused several times and is efficient, cost-effective, and eco-friendly. Moreover, the described protocol is easy and cheap to implement.

Keywords: 1-Amidoalkyl-2-naphthol, Phthalimide-*N*-sulfonic acid, Three-component reaction, Solvent-free.

INTRODUCTION

Multicomponent reactions (MCRs) have long been recognized as uniquely powerful tools to synthesis of structurally various chemical libraries of molecules through carbon-carbon and carbonheteroatom bond formations in a single reaction vessel from readily available starting materials without the isolation of intermediates. MCRs provided considerably higher efficiency and molecular complexity. They are usually associated with a number of advantageous such as green process of bond-forming, shorter reaction times, simplicity, avoidance of operational timeconsuming, energy and raw material saving, high bond-forming efficiency, minimal waste generation, reduction in the number of work-up, as well as no need for complicated purification processes [1-6].

Amidoalkyl naphthols can be used in pharmaceutical chemistry [7]. Trough amide hydrolysis reaction on the 1-amidoalkyl-2-naphthol derivatives, they can be converted into biologically useful compounds called 1-aminoalkyl-2-naphthols, which exhibit depressor and bradycardia effects [8]. 1-Amidoalkyl-2-naphthols can be also converted to derivatives of 1,3-oxazines as an exclusive class of bioactive compounds that present in many biologically important natural products and drug candidates [9, 10], which exhibit broad ranges of biological activities such as antihypertensive [11], analgesic [12], antirheumatic [13], antianginal [14], antibacterial, and antiviral [15].

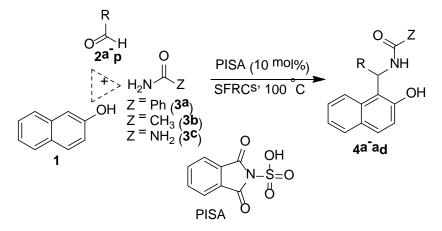
The synthesis of 1-amidoalkyl-2-naphthols using one-pot, three-component reaction (3CR) of various aldehydes, naphthols, and amide derivatives, one of the most effective and efficient reactions in the field of synthetic organic chemistry. In this context, a number of catalysts such as Lewis or Brönsted acids [16, 17], nanomaterials [18-24] and carbohydrates [25] have been used to catalysing this 3CR.

In recent years, catalytic systems containing sulfonic acid groups are used to synthesis of these compounds. Some of these catalysts include sulfanilic 1-methyl-3-(2acid [26], (sulfooxy)ethyl)-1*H*-imidazol-3-ium chloride [27], poly(4-vinylpyridinium butane sulfonic acid) hydrogen sulfate [28], polyethylene glycol (PEG)-based dicationic acidic ionic liquid $(PEG_{1000}-DAIL)$ [29], polymer supported sulphonic acid NKC-9 [30], 1,3-disulfonic acid imidazolium hydrogen sulfate{[Dsim]HSO₄} [31], heteropolyanion-based SO₃H [32], saccharin sulfonic acid [33], L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate (supported on silica gel) [34], β -cyclodextrin-butane sulfonic acid [35], and ionic liquid [36]. Sonochemically mediated silica chloride catalyst condensation has also been reported for the preparation of amidoalkyl naphthols [37]. However, some of the methods mentioned above, suffer from at least one of the following drawbacks: long reaction times, the use of organic solvents, forcing conditions, create relatively wastes, special instrumentation, expensive as well as toxic the reagents. Due to biological and pharmacological role of substances with amidoalkyl naphthol fragments, major attention has been focused on the development of convenient, efficient, inexpensive, and eco-friendly new methodologies using readily available reagents to synthesis of these types of valuable compounds.

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Scheme 1. One-pot three-component reaction (3-CR) of 2-naphthol (1), aryl aldehydes (2a-p), and amides (3a-c) in order to synthesis of 1-midoalkyl-2-naphthols (4a-ad) in the presence of PISA under SFRCs

Because many organic solvents are ecologically harmful, strategies for their minimized usage and developments toward benign chemical technologies are highly sought after [38]. From the view point of green chemistry, the exploration of safe methods with the aim to achieve greener, more sustainable, and environmentally friendly conditions is of eminent important. Among the ways to achieve this goal is the development of SFRs. They offer several advantages relative to using organic or other reaction media include: the compounds formed are often sufficiently pure, rapid, no need for specialized equipment, minimizing the energy consumption, avoidance of functional group protectiondeprotection. environmentally benign, costeffective, easy operation, high yields, and avoidance of pollution [39-43].

Phthalimid-*N*-sulfonic acid (PISA) has currently been synthesized and utilized by us as an efficient catalyst in synthesis of the Biginelli compounds [44]. In the present work, we report a rapid, green and easy procedure for the synthesis of 1amidoalkyl-2-naphthol derivatives (**4a-ad**) using PISA as the solid acidic organic catalyst under SFRCs (Scheme 1).

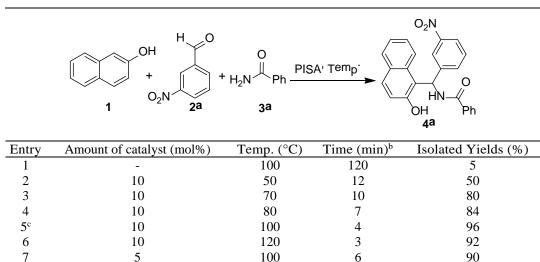
RESULTS AND DISCUSSION

In continuation of our investigations on synthesis of amidoalkyl naphthols [45], we decided to use the new catalyst for the MCR synthesis of the compounds mentioned above. At the beginning of the present investigation, phthalimid-*N*-sulfonic acid (PISA) was synthesized according to our previous work [44] and applied to preparation of a series of 1amidoalkyl-2-naphthols (**4a-ad**). In the direction of optimize the reaction conditions (amount of catalysts and reaction temperature) for preparation of 1amidoalkyl-2-naphthols (**4a-ad**), synthesis of *N*-((2hydroxynaphthalen-1-yl)(3-

nitrophenyl)methyl)benzamide (**4a**) was selected as the model product, and behaviour of its synthesis was explored in the presence of different amounts of PISA under SFRCs. The results are summarized in Table 1.

A test reaction for the synthesis of **4a** at 100 °C deprived of catalyst was implemented with the aim of establishes the efficacy of the catalyst. It was found that no product was observed even after 2 h heating (Table 1, entry 1). The product (4a) is formed neat 2-naphthol when (1), 3nitrobenzaldehyde (2a), benzamide (3a), and 10 mol % of PISA are mixed at 50 °C (Table 1, entry 2). Formation of product upon heating in the presence of a catalytic amount of PISA will further support a PISA for the achievement of products is essential. By increasing the reaction temperature from 50 to 70 and 80 °C, the reaction yield was improved, and the reaction time was shortened (Table 1, entries 3 and 4). The change in the yield from 84 %, to 96 % occurs when the temperature is increased from 80 °C to 100 °C (Table 1, entry 5). Further, at temperatures higher than 100 °C, the reaction did not proceed efficiently (Table 1, entry 6). In addition to the temperature of the reaction, the amount of the catalyst is also an important factor in the product formation. Hence, the effect of catalyst-loading on completion of the reaction at ambient temperature was also studied. The yield of the product was diminished and reaction time was also prolonged, when 5 mol % of catalyst was used (Table 1, entry 7). Higher amounts of the catalyst (i.e., 15 and 20 mol %) neither increased, nor lowered the yield% (Table 1, entries 8 and 9). Screening of the solvents such as EtOH, H₂O, CH₂Cl₂, EtOAc, and CH₃CN as the media reaction lead to low yields of product and prolonged reaction times compared with the SFRCs. Based on

Table 1. Screening the reaction conditions towards synthesis of N-((2-hydroxynaphthalen-1-yl)(3nitrophenyl)methyl)benzamide (4a)^a



100 ^a Reaction conditions: a well ground mixture of 1-naphthol 1 (1 mmol), 3-nitrobenzaldehyde 2a (1 mmol), benzamide 3a (1 mmol), and the catalyst was magnetically stirred.

100

3.5

2.5

^bProgress of the reaction was monitored by TLC analysis.

15

20

^c Optimized conditions shown in bold.

8

9

the above-mentioned studies, 100 °C and 10 mol % PISA was found to be the best optimal quantity and conditions, and satisfactory to push the reaction forward. Hence, these optimal conditions were applied to evaluate the generality of this procedure for the one-pot synthesis of the other amidoalkyl naphthols from 2-naphthol (1), a number of benzaldehydes (2a-m), heteroaryl aldehydes (2n-o), cinnamaldehyde (2p), and amides (3a-c), under SFRCs at 100 °C (Table 2).

Aromatic aldehydes carrying electron-donating (such as methyl or methoxy) or electronwithdrawing (such as nitro or halide) substituents reacted successfully in the presence of PISA as the catalyst and gave the corresponding products in excellent yields and shorter reaction times. It was also found that substituted benzaldehydes bearing electron-withdrawing groups (Table 2, entries 1-2, 4 and 6) gave higher yields and faster reacted than substituted benzaldehydes containing electrondonating groups (Table 2, entries 7-8, 10-12). When 3-methoxybenzaldehyd is used as substrate, possibly electron-withdrawing inductive effect is applied and most likely, this is one reason for increasing the yield and shortening reaction time (Table 2, entry 9). The reaction of sterically hindered ortho-substituted benzaldehydes including 2-chlorobenzaldehyde and 2-nitrobenzaldehyde were studied (Table 2, entries 3 and 5). In these cases, reaction time was longer and the yield of reaction was lower as compared to those of products from other benzaldehydes, which can be attributed to steric factors. The use of acetamide

(3b), urea (3c) instead of benzamide (3a) in the synthesis of the titled compounds, also gave similar results, as shown in Table 2 (entries 14-23 and 26-30). This approach was highly operative for the preparation of targeted compounds (4a-ad) as well as in all cases, 1-amidoalkyl-2-naphthols were the individual products and no by-product was observed. The reaction of 2-naphthol (1), acetamide (3a), and an aliphatic aldehyde (i.e., *n*-butyraldehyde) leads to trace amount of corresponding amidoalkyl naphthol product after one day. In addition, a reaction of 2naphthol (1) and acetamide (3a) with α,β unsaturated aldehydes such as cinnamaldehyde was implemented and lead to the formation of the product in good isolated yields (Table 2, entry 26). The reaction of 2-naphthol (1), and acetamide (3a) with hetero-aromatic aldehydes [for example thiophene-2-carbaldehyde (**2n**) and furan-2carbaldehyde (20)] leads to corresponding products with 79 and 75 % yields, respectively (Table 2, entries 24-25).

95

92

The reusability of the catalyst was also investigated in the model reaction under optimized reaction conditions (Table 3). Upon completion, the catalyst was recovered after each run and reused for the same reaction. It showed nearly the same activity as a catalyst along but with a slight decrease of yields. The procedure was repeated and the results indicated that the catalyst could be recycled five times with only a slight loss of catalytic activity. This indicated that the PISA was an efficient and H. Kiyani, H. Darbandi: One-pot three-component synthesis of 1-amidoalkyl-2-naphthols in the presence of phthalimide-N-sulfonic...

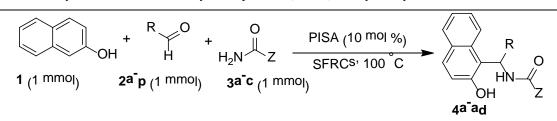
recyclable catalyst for the preparation of 1-amidoalkyl-2-naphthol derivatives.

Mechanistically (Scheme 2), formation of 1amidoalkyl-2-naphthol products (4a-ad) could proceed *via* formation of *ortho*-quinone methides [31-33] between 2-naphthol (1) and the aldehydes (2), assisted by PISA. Then, the Michael addition of amides (3) to the *ortho*-QMs delivers the 1amidoalkyl-2-naphthols. The SO₃H functional group of PISA could provide acidic site for activating aldehydes efficiently, so facilitating this 3-CR.

In order to display the applicability and comparison the efficacy of the PISA catalyst with

other catalysts in the preparation of 1-amidoalkyl-2naphthols, the obtained results in the synthesis of 4a tabulated in Table 4. Compared with some of those reported in the literature, this method is comparable to the formerly reported methods in terms of reaction times and product yields. It can be seen that, PISA is also an equal or more efficient catalyst for this 3-CR. Contrary to some of the pervious reported methods, this work does not require any ionic liquids, hazardous solvents such as chloroform or special devices such as ultrasound and microwave.

Table 2. The 3C synthesis of 1-amidoalkyl-2-naphthols (4a-ad) catalysed by PISA under SFRCs at 100 °C



Entr	R, Aldehyde	Ζ	Product	Time	Isolated	Mp (°C)	
У	•			(min)	yields	Obs.	Rep. [ref.]
					(%)		-
1	3-NO ₂ -C ₆ H ₄ , 2a	Ph, 3a	4 a	4	96	238-240	234-236 [24]
2	4-NO ₂ -C ₆ H ₄ , 2b	Ph, 3a	4b	5	92	239-241	238-240 [24]
3	$2-NO_2-C_6H_4$, 2c	Ph, 3a	4 c	9	88	261-263	262-264 [24]
4	4-Cl-C ₆ H ₄ , 2d	Ph, 3a	4d	6	90	186-187	186-188 [24]
5	2-Cl-C ₆ H ₄ , 2e	Ph, 3a	4e	8	87	198-200	263-265 [24]
6	4-F-C ₆ H ₄ , 2f	Ph, 3a	4f	5	90	194-195	191-193 [16]
7	4-CH ₃ -C ₆ H ₄ , 2g	Ph, 3a	4g	8	88	216-217	215-216 [24]
8	4-CH ₃ O-C ₆ H ₄ , 2h	Ph, 3a	4h	8	90	207-209	208-211 [24]
9	3-CH ₃ O-C ₆ H ₄ , 2i	Ph, 3a	4i	5	94	231-233	232 [20]
10	2,5-(CH ₃) ₂ O-C ₆ H ₃ , 2 j	Ph, 3a	4j	7	92	237-239	238-240 [46]
11	2,4-(CH ₃) ₂ O-C ₆ H ₃ , 2k	Ph, 3a	4 k	8	90	228-230	227-229 [9]
12	3-CH ₃ O-4-OH-C ₆ H ₃ , 2 I	Ph, 3a	41	8	94	218-220	219 [33]
13	C ₆ H ₅ , 2m	Ph, 3a	4 m	5	91	237-238	237-239 [16]
14	3-NO ₂ -C ₆ H ₄ , 2a	CH ₃ , 3b	4n	5	92	242-243	241-242 [28]
15	4-NO ₂ -C ₆ H ₄ , 2b	CH ₃ , 3b	4o	5	92	244-246	243-245 [28]
16	$2-NO_2-C_6H_4$, 2c	CH ₃ , 3b	4p	10	86	181-182	182 [18]
17	4-Cl-C ₆ H ₄ , 2d	CH ₃ , 3b	4 q	7	89	225-227	226-228 [28]
18	2-Cl-C ₆ H ₄ , 2e	CH ₃ , 3b	4 r	8	87	209-211	210-211 [27]
19	4-F-C ₆ H ₄ , 2f	CH ₃ , 3b	4 s	12	84	208-209	206-208 [30]
20	4-CH ₃ -C ₆ H ₄ , 2g	CH ₃ , 3b	4t	10	88	220-222	221-223 [28]
21	4-CH ₃ O-C ₆ H ₄ , 2h	CH ₃ , 3b	4 u	10	87	181-183	181-183 [28]
22	3-CH ₃ O-C ₆ H ₄ , 2i	CH ₃ , 3b	4 v	6	97	204-206	203-205 [28]
23	C ₆ H ₅ , 2m	CH ₃ , 3b	4 w	5	90	242-243	242-244 [28]
24	2-Thienyl, 2n	CH ₃ , 3b	4x	10	79	223-224	222-224 [26]
25	2-Furyl, 20	CH ₃ , 3b	4 y	12	75	218-220	218-220 [47]
26	С ₆ Н ₅ —СН=СН, 2р	CH3, 3b	4z	18	88	175-177	174.5-176 [19]
27	$3-NO_2-C_6H_4$, 2a	NH ₂ , 3c	4aa	7	93	193-195	192-194 [24]
28	4-NO ₂ -C ₆ H ₄ , 2b	NH ₂ , 3c	4ab	9	94	191-193	192-194 [24]
29	4-Cl-C ₆ H ₄ , 2d	NH ₂ , 3c	4ac	6	91	165-167	166-168 [24]
30	C ₆ H ₅ , 2m	NH ₂ , 3c	4ad	12	92	176-177	176-178 [24]

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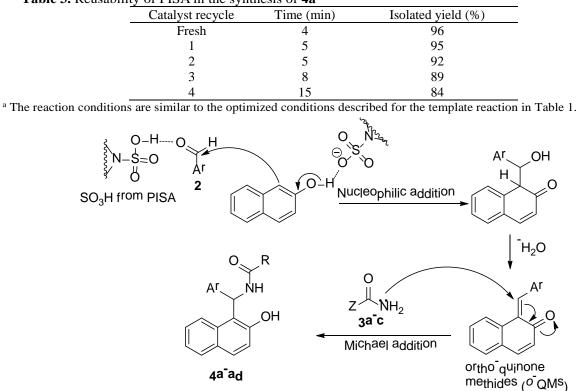


Table 3. Reusability of PISA in the synthesis of 4a^a

Scheme 2. Proposed mechanism for synthesis of 1-amidoalkyl-2-naphthols (4a-ad)

Table 4. Comparison of the catalytic performance of PISA for the one-pot 3C synthesis of **4a** with those obtained by reported catalysts.

Entry	Catalyst (mol %) [g]/conditions [ref.]	Time (min)	Yield (%)
1	[Msim]Cl (10)/SF, 120 °C [16]	5	90
2	Maltose (20)/SF, 100 °C [25]	18	90
3	Sulfanilic acid (20)/SF, MW [26]	8	89
4	Sulfanilic acid (20)/SF, 110-120 °C [26]	14	88
5	MSI (6)/ionic liquid [Bpy]BF4, 80 °C [27]	25	95
6	PEG ₁₀₀₀ -DAIL (0.03 mmol)/80 °C [29]	5	94
7	[MIMPS]H ₂ PMo ₁₂ O ₄₀ (10)/SF, 110 °C [32]	5	93
8	Silica chloride [0.1]/US, 28-30 °C [34]	9	98
9	[Dsim]HSO ₄ (5)/SF, 80 °C [31]	20	97
10	PSSA-NKC-9 [0.17]/chloroform, reflux [30]	360	88
11	2-HSBA (10)/SF, 100 °C [45]	5	95
12	PISA (10)/SF, 100 °C [Present work]	4	96

[*Msim*]*Cl*, 3-methyl-1-sulfonic acid imidazolium chloride; *SF*, solvent-free; *MSI*, 1-methyl-3-(2-(sulfooxy)ethyl)-1*H*imidazol-3-ium chloride; [*Bpy*]*BF*₄, *N*-butylpyridinium tetrafluoroborate; *PEG*₁₀₀₀-*DAIL*, PEG-based dicationic acidic ionic liquid; *MIMPS*, *N*-methyl-imidazolium propane sulphonate; [*Dsim*]*HSO*₄, 1,3-disulfonic acid imidazolium hydrogen sulfate; *PSSA*-*NKC*-9, Polymer supported sulphonic acid NKC-9; *US*, ultrasond; 2-*HSBA*, 2-hydroxy-5-sulfobenzoic acid.

CONCLUSIONS

In summary, an efficient, simple, and environmentally benign protocol towards synthesis of 1-amidoalkyl-2-naphthols has been developed. Thermal SFRCs were applied to afford the corresponding 1-amidoalkyl-2-naphthol compounds. PISA display excellent catalytic activity toward this 3CR. The use of PISA in this 3CR is included merits such as good to high yields, short reaction times, practical simplicity, recyclable catalyst, clean formation of the target products, no use of solvent, and easy work-up.

EXPERIMENTAL

All chemicals were purchased from Alfa Aesar and Aldrich as well as were used without further purification, with the exception of 4methylbenzaldehyde, 4-methoxylbenzaldehyde, benzaldehyde, and thiophene-2-carbaldehyde which were distilled before using. All solvents were distilled before using. 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 and are uncorrected. NMR spectra were recorded at ambient temperature on a BRUKER AVANCE DRX-400 MHz using CDCl₃ or 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 precoated silica gel 60 F254 aluminum sheets, visualized by UV light.

General procedure for synthesis of 1-amidoalkyl-2naphthols (**4a-ad**)

A mixture of 2-naphthol 1 (1 mmol), aldehyde 2 (1 mmol), amide 3 (benzamide, acetamide, or urea 1 mmol) and PISA (10 mol %) was stirred at 100 °C in an oil bath for 4-18 min. After completion of the reaction (using TLC analysis), the reaction mixture was allowed to cool to room temperature. After that, the products was extracted by hot ethyl acetate (the product is soluble in hot ethyl acetate, but PISA is not soluble in this solvent), and then the catalyst was recovered and washed thoroughly with ethyl acetate and then diethyl ether. After being dried, it was subjected to another reaction with the same substrates. Pure 1-amidoalkyl-2-naphthols were afforded by evaporation of the solvent followed by recrystallization from ethanol. The selected spectral data for representative compounds (4a and 4m) as follows:

N-((2-Hydroxynaphthalen-1-yl)(3-

nitrophenyl)methyl)benzamide (**4a**) IR (KBr, cm⁻¹): 3374, 3262, 3055, 2974, 1633, 1530, 1504, 1475, 1440, 1345, 1253, 1145, 735; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 10.42 (s, 1H), 9.15 (d, *J* = 8.0 Hz, 1H), 8.11-8.09 (m, 3H), 7.91-7.84 (m, 4H), 7.72 (d, *J* = 7.5 Hz, 1H), 7.60-7.49 (m, 5H), 7.41 (d, *J* = 8.0 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 1H), 7.25 (d, *J* = 8.5 Hz, 1H); ¹³C NMR (100 MHz, DMSO-*d*₆): δ = 166.8, 153.9, 148.2, 145.1, 134.4, 133.7, 132.8, 132.1, 130.5, 130.3, 129.3, 128.9, 128.8, 127.9, 127.6, 123.4, 122.9, 122.2, 121.3, 119.2, 117.3, 49.5.

N-((2-Hydroxynaphthalen-1-yl)(3-

nitrophenyl)methyl)acetamide (4m) IR (KBr, cm⁻¹): 3395, 3151, 3090, 2989, 1645, 1530, 1435, 1348, 1295, 1279, 1071, 990, 828, 745, 734; ¹H NMR (400 MHz, DMSO- d_6): $\delta = 10.14$ (s, 1H), 8.62 (d, J = 8.0Hz, 1H), 8.02-7.99 (m, 2H), 7.84 (br, 1H), 7.78 (t, J = 8.6 Hz, 2H), 7.59-7.51 (m, 2H), 7.40 (t, J = 7.6 Hz, 1H), 7.26 (t, J = 7.4 Hz, 1H), 7.18 (d, J = 8.7 Hz, 1H), 7.16 (t, J = 8.0 Hz, 1H), 2.02 (s, 3H); ¹³C NMR (100 MHz, DMSO- d_6): $\delta = 170.3$, 153.9, 148.2, 145.9, 133.4, 132.7, 130.5, 130.1, 129.2, 128.9, 127.3, 123.2, 123.1, 121.8, 120.9, 118.9, 118.3, 48.2, 23.1.

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ЕДНО-СТАДИЙНА ТРИ-КОМПОНЕНТНА СИНТЕЗА НА 1-АМИДОАЛКИЛ-2-НАФТОЛИ В ПРИСЪСТВИЕ НА ФТАЛИМИД-*N*-СУЛФОНОВА КИСЕЛИНА

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(Резюме)

Изпитана е проста, едно-стадийна три-компонентна реакция (3CR) за синтезата на различни 1-амидоалкил-2-нафтоли при фталимид-*N*-сулфонова киселина като твърд органичен катализатор. Реакцията се провежда без разтворител при 100°C за около 4-18 минути, а съответните продукти са с добиви между 75 и 97%. Катализаторът се възстановява и се използва няколко пъти. Той е ефективен, евтин и екологически съвместим. Освен това описаният протокол е лесен и евтин за изпълнение.