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Multicomponent synthesis of bioactive 1-amidoalkyl-2-naphtols under solvent-free conditions

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A one-pot condensation reaction of 2-naphthol, aromatic aldehydes, and acetamide or thioacetamide catalyzed by L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate (supported on silica gel) to afford 1-amidoalkyl-2-naphthols under solvent-free conditions is described. The advantages of this method are good yields, easy work-up, mild and solvent-free conditions and inexpensive catalyst.

Keywords: L-Pyrrolidine-2-carboxylic acid-4-hydrogen sulfate; Multicomponent reaction; Amidoalkyl naphtols; Solvent-free.

INTRODUCTION

Multicomponent reaction (MCR) condensations involve three or more simple substrates reacting in a single event to create structurally complex organic compounds which contain the fundamental parts of all the starting materials [1]. It is considered that multicomponent reactions approach close to the "ideal synthesis" due to atom economy, and high bond forming efficiency [2]. Therefore, chemists have made great efforts to find and develop new MCRs.

Compounds containing 1,3-amino oxygenated functional groups such as 1-amidoalkyl-2-naphthol derivatives are frequently found in a variety of biologically active natural products and potent drugs such as a number of nucleoside antibiotics and HIV protease inhibitors [3-5]. Furthermore, these compounds can be converted to important bioactive 1-aminomethyl-2-naphthols by amide hydrolysis reaction. These compounds exhibit hypotensive, bradycardia effects and Ca^{2+} channel-blocking activities in humans [6,7].

1-amidoalkyl-2-naphthols can be prepared by three-component condensation of 2-naphthol, aldehydes, and different amides in the presence of Brønsted or Lewis acidic catalysts such as PEG-based dicationic acidic ionic liquid [8], $Al(HSO_4)_3$ [9], $Fe(HSO_4)_3$ [10], H_3BO_3 [11], [TEBSA][HSO_4] [12], cation exchange resins [13], Cu-exchanged heteropoly acids [14], dodecylphosphonic acid [15], N,N,N',N'-tetrabromobenzene-1,3-disulfonamide [16], $RuCl_2(PPh_3)_3$ [7], $H_3PW_{12}O_{40}$ [17], [Bmim]Br [18], $[NMP]^+HSO_4^-$ [19], molybdophosphoric acid [20], imidazolium salts [21], $Cu(ClO_4)_2.6H_2O$ [22], tin tetrachloride [23], sulfamic acid [24], zinc benzenesulfonate [25], iodine [26], montmorillonite K10 [27], graphite supported perchloric acid [28], trityl chloride [29], MCM-41-*N*-propylsulfamic acid [30]. However, some of the reported methods suffer from disadvantages such as prolonged reaction time, low yields, use of toxic solvent, use of large amount of catalyst, strongly acidic conditions, microwave or ultrasonic irradiation and harsh reaction conditions. Therefore, finding a versatile, green and simple protocol that uses a highly efficient catalyst for preparation of amidoalkyl naphthols is still desirable.

In this paper, we report a simple and efficient procedure for one-pot synthesis of 1-amidoalkyl-2-naphthols from 2-naphthol, aromatic aldehydes and acetamide/thioacetamide in the presence of catalytic amounts of L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate (supported on silica gel) under solvent-free conditions.

RESULTS AND DISCUSSION

In continuation of our research with heterogeneous catalysts [30-34], herein we report a mild, simple and effective procedure for one-pot synthesis of 1-amidoalkyl-2-naphthol derivatives *via* a multi-component condensation reaction between aryl aldehydes, 2-naphthol and acetamide or thioacetamide in the presence of L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate (supported on silica gel) at 100 °C (scheme 1).

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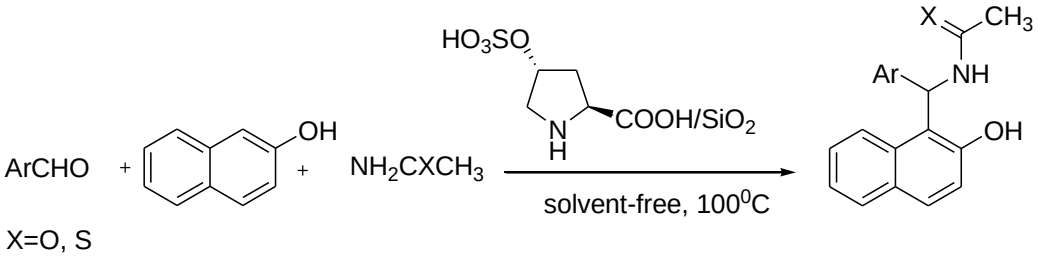


Scheme 1. Synthesis of 1-amidoalkyl-2-naphthols

In initial experiments to find the optimum conditions, a model reaction of 4-bromobenzaldehyde (1 mmol), 2-naphthol (1 mmol), and acetamide (1.6 mmol) in the presence of supported L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate on silica gel was performed under solvent-free conditions at 100 °C (Table 1). In the absence of any catalyst, the yield was low (entry 2), whereas good results were obtained in the presence of 0.1 mmol of catalyst (entry 1). In all cases the amount of the catalyst was crucial to a good yield and reaction rate. Also the scope of acetamide was studied (entry 3 and 4).

Next, to determine the effect of temperature on the reaction, the reaction of 4-bromobenzaldeyde, 2-naphthol, and acetamide in the presence of supported L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate on silica gel was performed under solvent-free conditions at different temperatures in an oil bath. As shown in table 2 the best result was achieved at 100 °C.

Table 2. Effect of temperature on the synthesis of N-[(4-bromo-phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl]-acetamide in the presence of L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate (supported on silica gel) under solvent-free conditions

Entry	Temperature (C°)	Yield (%)[b]
1	25	-[c]
2	40	-[d]
3	50	34
4	60	61
5	70	70
6	80	77
7	90	81
8	100	94

[a] 4-bromobenzaldehyde/2-naphtol/acetamide/catalyst = 1:1:1.6:0.1 mmol.
[b] yields refer to pure isolated products. [c] No reaction. [d]Trace conversion.

In all reactions, good to excellent yields were obtained at appropriate reaction times. Clean and complete conversions leading to the corresponding 1-amidoalkyl-2-naphthols were observed. Aromatic aldehydes carrying either electron-withdrawing (nitro) or electron- donating (halide, alkyl, alkoxyl) groups were all suitable for the reactions.

The proposed mechanism for this acid catalysis condensation reaction *via in situ* generation of ortho-quinone methides (o-QMs), is presented in Scheme 2. The o-QMs have been reacted with acetamide or thioacetamide *via* conjugate addition to form 1-amidoalkyl-2-naphtols as final products.

In comparison with other catalysts employed for the synthesis of N-[(4-methyl-phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl] acetamide from 4-methylbenzaldehyde, 2-naphthol and acetamide, L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate (supported on silica gel) showed significant catalytic activity in terms of good reaction time, high yield and mild conditions (table 3).

In conclusion, supported L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate on silica gel as a green and inexpensive catalyst can efficiently catalyze the synthesis of 1-amidoalkyl-2-naphthols *via* one-pot three-component reaction of 2-naphthol, acetamide /thioacetamide and aromatic aldehydes. This solvent-free procedure has advantages such as mild reaction conditions, straightforward workup and purification, excellent yields and environmental benignancy.

EXPERIMENTAL

Materials and Methods

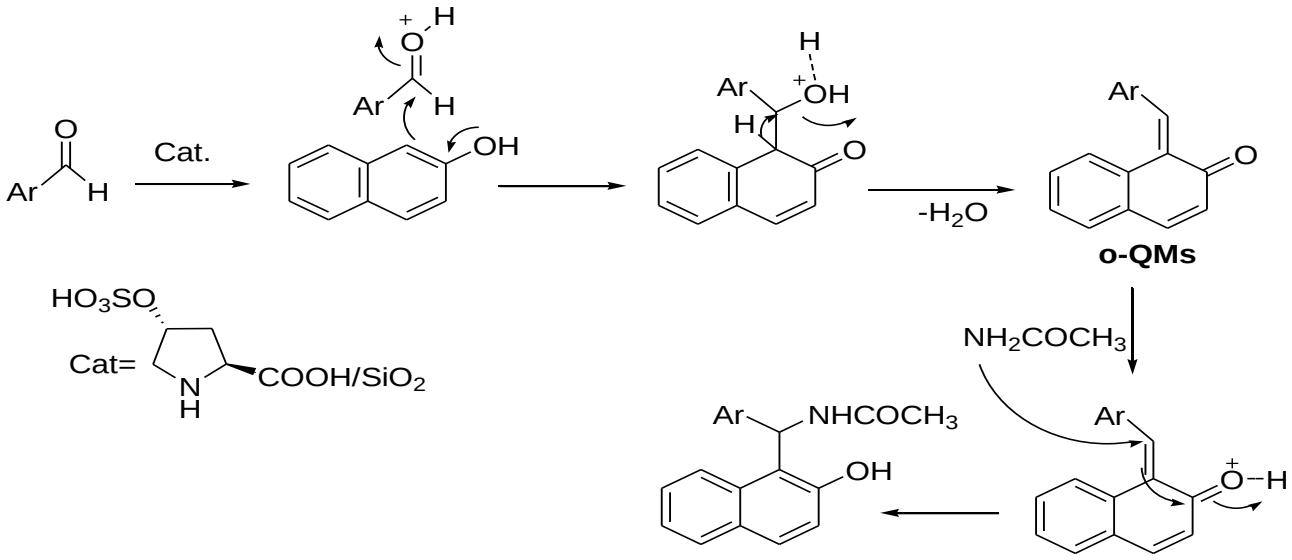
All chemicals were purchased from Fluka, Merck and Aldrich chemical companies. The products were characterized by comparison of their spectral (^{1}H NMR, and ^{13}C NMR) and physical data with those of authentic samples.

Supported L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate on silica gel as a solid acid catalyst is safe and easy to handle catalyst. This catalyst was prepared by the reaction of L-hydroxyproline with chlorosulfonic acid at room temperature according to the reported procedure [35]. Because of the gummy properties of L-pyrrolidine-2-carboxylicacid-4-hydrogensulfate, it was supported on silica gel.

Table 1. The one-pot three-component reaction of 2-naphthol, acetamide/thioacetamide and aryl aldehydes in the presence of L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate (supported on silica gel) under solvent-free conditions at 100 °C[a]

Entry	Aldehyde	amide	Time(h)	Yield(%)[b]	m.p. (lit. m.p.)	Ref.
1	4-Bromobenzaldehyde	CH_3CONH_2	3	94	235.5-236.7 (229-231)	[12]
2	4-Bromobenzaldehyde	CH_3CONH_2	3	22[c,f]	234-236 (229-231)	[12]
3	4-Bromobenzaldehyde	CH_3CONH_2	5	86[d]	235-237 (229-231)	[12]
4	4-Bromobenzaldehyde	CH_3CONH_2	5	90[e]	235-236.5 (229-231)	[12]
5	4-Chlorobenzaldehyde	CH_3CONH_2	2	90	231.5-232.9 (229-230)	[12]
6	4-Flourobenzaldehyde	CH_3CONH_2	6	84	223-224 (230-232)	[9]
7	3-Nitrobenzaldehyde	CH_3CONH_2	1.75	95	241-243 (241-242)	[8]
8	2-Nitrobenzaldehyde	CH_3CONH_2	1.66	91	215-216 (218-219)	[36]
9	4-Methylbenzaldehyde	CH_3CONH_2	2	93	220-221 (221-223)	[8]
10	4-Methoxybenzaldehyde	CH_3CONH_2	5.5	91	182-183 (181-183)	[8]
11	2-Naphthaldehyde	CH_3CONH_2	2.6	94	218-220 (220-222)	[11]
12	4-Hydroxybenzaldehyde	CH_3CONH_2	4	92	224.5-225.5	-
13	5-Bromo-2-hydroxybenzaldehyde	CH_3CONH_2	6	95	212.5-213	-
14	4-Bromobenzaldehyde	CH_3CSNH_2	7	83	214.5-215.5	-
15	4-Chlorobenzaldehyde	CH_3CSNH_2	5.5	89	214-215	-
16	3-Nitrobenzaldehyde	CH_3CSNH_2	6	83	231-232	-
17	4-Methylbenzaldehyde	CH_3CSNH_2	6	84	199-201	-
18	5-Bromo-2-hydroxybenzaldehyde	CH_3CSNH_2	7	45[f]	216.5-217.5	-

[a]reaction conditions: aldehyde/2-naphtol/acetamide or thioacetamide/catalyst= 1:1:1.6:0.1 mmol. [b] yields refer to pure isolated products. [c] reaction is performed in the absence of catalyst. [d] amount of acetamide was 1.2 mmol. [e] amount of acetamide was 1.4 mmol. [f] yield determined by preparative TLC.



Scheme 2. Plausible mechanism of the one-pot synthesis of 1-amidoalkyl-2 naphthols -

Table 3. Comparative results of L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate (supported on silica gel) with other catalysts for the synthesis of N-[(4-methyl-phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl]-acetamide

Entry	Catalyst (mol %)	Time (h)	Yield (%)	Ref.
1	Molybdophosphoric acid (0.12 g)	4	92	[20]
2	$Al(HSO_4)_3$ (0.05 g)	9 min	83	[9]
3	Graphite-$HClO_4$ (7.5)	2	74	[28]
4	Polymer-supported sulphonic acid (0.17 g)	4	92	[37]
5	Cu-exchanged heteropoly acids (5)	1.5	85	[14]
6	MCM-41-*N*-propylsulfamic acid (0.1 g)	3	95	[30]
7	L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate (0.0423 g)	2	93	This work

General procedure: solvent-free synthesis of 1-amidoalkyl-2-naphthols using supported L-pyrrolidine-2-carboxylic acid-4-hydrogen sulfate on silica gel as catalyst at 100 °C

In a typical reaction, to a mixture of 2-naphthol (1 mmol), aldehydes (1 mmol) and acetamide/thioacetamide (1.6 mmol), catalyst (0.1 mmol, 0.0423 g) was added. The mixture was stirred at 100 °C in an oil bath as indicated by thin-layer chromatography (TLC) for a complete reaction. After completion of the reaction, the mixture was cooled to room temperature, then the solid residue was dissolved in boiling EtOH and the mixture was stirred for 10 min. The pure product was obtained by recrystallization from water and ethanol (50:50).

The spectral data of some representative amidoalkyl naphthols are given below:

N-[(5-bromo-2-hydroxy-phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl]-acetamide (Table 1, Entry 13) ^{1}H NMR (400 MHz, DMSO-d_6): δ_H =1.92 (s, 3H), 6.66 (d, *J* = 8.8, 1H), 7.13-7.20 (m, 3H), 7.28 (m, 1H), 7.44 (m, 1H), 7.60 (s, 1H), 7.71 (d, *J* = 8.8, 1H), 7.78 (d, *J* = 8, 1H), 8.20 (d, *J* = 8.4, 1H), 8.45 (d, *J* = 8.4, 1H), 9 .73- 9.90 (br, 2H) (ppm). ^{1}H-NMR (400 MHz, DMSO+ D_2O): δ_H= 1.89 (s, 3H), 6.69 (d, *J* = 8.8, 1H), 7.11-7.18 (m, 3H), 7.26 (m, 1H), 7.39-7.42 (m, 1H), 7.47 (s, 1H), 7.70(d, *J* = 8.8, 1H), 7.76 (d, *J* = 7.6, 1H), 8.10 (d, *J* = 8.4, 1H) (ppm). ^{13}C-NMR (100 MHz, DMSO): δ_C= 23.1, 44.9, 110, 117.4, 119, 119.1, 122.7, 123.7, 126.5, 128.6, 128.8, 129.3, 130.3, 131.7, 131.8, 133.1, 153.7, 154.3, 169 (ppm).

N-[(4-bromo-phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl]-acetamide (Table 1, Entry 1) ^{1}H NMR (400 MHz, DMSO-d_6): δ_H= 2 (s, 3H), 7.10 (m, 3H), 7.23-7.30 (m, 2H), 7.38 (d, *J* = 8, 1H), 7.45 (d, *J* = 8.4, 2H), 7.80 (m, 3H), 8.49 (d, *J* = 8, 1H), 10.07 (s,1H) (ppm). ^{1}H-NMR (400 MHz, DMSO+ D_2O): δ_H= 1.98 (s, 3H), 7.07 (m, 3H), 7.20(d, *J* = 8.8, 1H), 7.26 (m, 1H), 7.38 (m, 3H), 7.77 (m, 3H) (ppm).

N-[(4-chloro-phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl]-acetamide (Table 1, Entry 5) ^{1}H NMR (400 MHz, DMSO-d_6): δ_H= 2 (s, 3H), 7.10 (d, *J* =8, 1H), 7.17 (d, *J* = 8.4, 2H), 7.23 (d, *J* = 8.8, 1H), 7.26-7.33 (m, 3H), 7.39 (m, 1H), 7.78-7.83 (m, 3H), 8.48 (d, *J* = 8, 1H), 10.06 (s, 1H) (ppm). ^{13}C-NMR (100 MHz, DMSO): δ_C= 23.1, 56.6, 118.9, 119, 123, 123.6, 127, 128.4, 128.9, 129.1, 130, 131.1, 132.7, 142.3, 153.7, 170 (ppm).

N-[(4-fluoro-phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl]-acetamide (Table 1, Entry 6) ^{1}H NMR (400 MHz, DMSO-d_6): δ_H= 2 (s, 3H), 7.06-7.12 (m, 3H), 7.17-7.3 (m, 4H), 7.39 (m, 1H), 7.77-7.83 (m, 3H), 8.49 (d, *J* = 8.4, 1H), 10.05 (s, 1H) (ppm). ^{13}CNMR (100 MHz, DMSO-d_6): δ_C= 22.6, 47.33, 114.51, 118.2, 122.41, 122.8, 126.38, 128.0, 128.55, 129.54. 132.19, 138.6, 153.12, 159.48, 161.88, 169.26 (ppm)

*N-[(3-nitro-phenyl)-(2-hydroxy-napthalen-1-yl)-methyl]-acetamide (*Table 1, Entry 7*)* ^{1}H NMR (400 MHz, DMSO-d6): δ_H= 2.04 (s, 3H), 7.27 (m, 3H), 7.43 (t, *J* = 7.2, 1H), 7.57 (m, 2H), 7.86 (m, 3H), 8.03 (s, 1H) 8.06 (m, 1H), 8.65 (d, *J* = 8, 1H), 10.17 (s, 1H) (ppm). ^{13}C-NMR (100 MHz, DMSO): δ_C= 23, 48.1, 118.3, 118.9, 120.9, 121.7, 123.1, 123.3, 127.3, 128.9, 129.2, 130.1, 130.4, 132.6, 133.3, 145.9, 148.2, 153.8, 170.2 (ppm).

*N-[(4-methyl-phenyl)-(2-hydroxy-napthalen-1-yl)-methyl]-acetamide (*Table 1, Entry 9*)* ^{1}HNMR (400 MHz, DMSO-d_6): δ_H= 1.99 (s, 3H), 2.24 (s, 3H), 7.1 (s, 1H), 7.23 (d, 1H), 7.24 (d, 4H), 7.26 (m, 1H), 7.75 (m, 1H), 7.78 (d, 1H), 7.79 (d, 1H), 7.82 (d, 1H), 8.44 (s,1H), 9.98 (s,1H) (ppm) ^{13}CNMR (100 MHz, DMSO-d_6): δ_C= 47.61, 118.46, 118.98, 122.32, 125.97, 126.21, 129.1, 128.51, 132.3, 134.9, 139.55, 153.07, 169.14 (ppm)

N-[(5-bromo-2-hydroxy-phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl]- thioacetamide (Table 1, Entry 18) ^{1}H NMR (400 MHz, DMSO-d_6): δ_H= 1.92 (s, 3H), 6.66 (d, *J* = 8.8, 1H), 7.12-7.20 (m, 3H), 7.27 (m, *J* = 14.8, 1H), 7.44 (m, *J* = 14.8, 1H), 7.60 (s, 1H), 7.71 (d, *J* = 8.8, 1H), 7.78 (d, *J* = 8, 1H), 8.20 (d, *J* = 8.8, 1H), 8.44 (d, *J* = 8.4, 1H), 9 .70 (s, 1H), 9.91 (s, 1H) (ppm). ^{1}H-NMR (400 MHz, DMSO+ D_2O): δ_H= 1.88 (s, 3H), 6.68 (d, *J* = 8.8, 1H), 7.10-7.18 (m, 3H), 7.27 (m, 1H), 7.39- 7.46 (m, 2H), 7.71 (d, *J* = 8.8, 1H), 7.76 (d, *J* = 8, 1H), 8.10 (d, *J* = 8.8, 1H) (ppm). ^{13}C-NMR(100 MHz, DMSO): δ_C= 23.1, 44.9, 110, 117.4, 119, 119.1, 122.7, 123.7, 126.5, 128.6, 128.8, 129.3, 130.3, 131.7, 131.8, 133.1, 153.7, 154.3, 169 (ppm).

N-[(4-bromo-phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl]-thioacetamide (Table 1, Entry 14) ^{1}H NMR (400 MHz, DMSO-d_6): δ_H= 2 (s, 3H), 7.10 (m, 3H), 7.23-7.30 (m, 2H), 7.38 (d, *J* = 7.6, 1H), 7.45-7.47 (m, 2H), 7.80 (m, 3H), 8.49 (d, *J* = 8.4, 1H), 10.07 (s,1H) (ppm). ^{1}H-NMR (400MHz, DMSO+ D_2O): δ_H= 1.98 (s, 3H), 7.1-7.6 (m, 3H), 7.21 (m, 1H), 7.24-7.28 (m, 1H), 7.39-7.43 (m, 3H), 7.76- 7.80(m, 3H) (ppm). ^{13}C-NMR (100 MHz, DMSO): δ_C= 23.1, 47.9, 118.8, 118.9, 119.5, 123, 123.7, 127, 128.8, 129, 129.1, 130, 131.3, 132.7, 142.7, 153.7, 170(ppm).

N-[(3-nitro-phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl]-thioacetamide (Table 1, Entry 14) ^{1}H NMR (400 MHz, DMSO-d_6): δ_H = 2.04 (s, 3H), 7.19-7.25 (m, 2H), 7.31 (m, 1H), 7.43 (m, 1H), 7.55-7.60 (m, 2H), 7.82-7.86 (m, 4H), 8.03-8.08 (m, 2H), 8.65 (d, *J* = 8, 1H), 10.16 (s, 1H) (ppm). ^{1}H-NMR (400 MHz, DMSO+D_2O): δ_H= 2.01 (s, 3H), 7.15 (s, 1H), 7.2 (d, *J* = 8.8, 1H), 7.3 (m, 1H), 7.42 (m, 1H), 7.54 (m, 1H), 7.6 (d, *J* = 8, 1H), 7.79- 7.87 (m, 2H), 7.9 (s, 1H), 8.02-8.04 (m, 1H) (ppm).^{13}C-NMR(100 MHz, DMSO): δ_C= 23, 48, 118.3, 118.9, 120.9, 121.7, 123.1, 123.3, 127.2, 128.9, 129.2, 130.1, 130.4, 132.6, 133.3, 145.9, 148.2, 153.8, 170.2 (ppm).

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МНОГОКОМПОНЕНТНА СИСТЕМА НА БИОАКТИВНИ 1-АМИДОАЛКИЛ-2-НАФТОЛИ В ОТСЪСТВИЕ НА РАЗТВОРИТЕЛ

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(Резюме)

Описана е едностадийна реакция на кондензация на 2-нафтол, ароматни алдехиди с ацетамид или тиоацетамид, катализирана от L-пиролидин-2-карбоксилова киселина-4-хидроген сулфат (нанесен върху силикагел) за получаването на 1-амидоалкил-2-нафтоли в отсъствие на разтворител. Предимствата на метоода са добрите добиви, лесното изпълнение, меките условия, отсъствието на разтворител и евтиния катализатор.