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Eco-friendly and efficient multi-component method for preparation of 1-amidoalkyl-2-naphthols under solvent-free conditions by dodecylphosphonic acid (DPA)

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ABSTRACT

An efficient and direct eco-friendly protocol for the preparation of 1-amidoalkyl-2-naphthols employing a multi-component, one-pot condensation reaction between β -naphthol, aromatic or aliphatic aldehydes and benzamide or acetamide in the presence of dodecylphosphonic acid under solvent-free conditions has been described.

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1. Introduction

Multi-component reactions (MCRs) play an important role in combinatorial chemistry because of their ability to synthesize small drug-like molecules with several degrees of structural diversity. These reactions allow compounds to be synthesized in few steps and usually in a one-pot manner [1]. Being time and energy saving while having simple procedures, high bond forming efficiency and low expenditures are among the advantages of these reactions [2]. Therefore finding and designing new MCRs has been the subject of extensive research.

Compounds containing 1,3-amino-oxygenated functional groups are frequently found in biologically active natural products and potent drugs such as nucleoside antibiotics and HIV protease inhibitors [3–6]. Furthermore, 1-amidoalkyl-2-naphthols can be converted to useful and important biological building blocks and to 1-aminomethyl-2-naphthols, which exhibit depressor

effects and bradycardia [7,8], by an amide hydrolysis reaction.

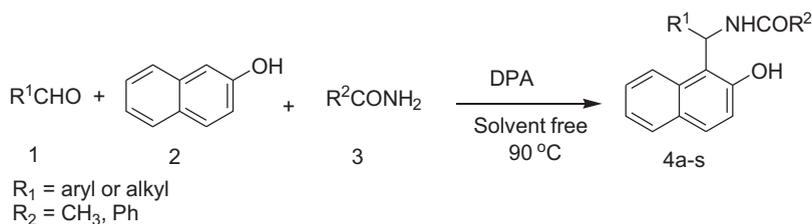
The preparation of 1-amidoalkyl-2-naphthols can be carried out by condensation of aryl aldehydes, β -naphthol, and acetonitrile or amides in the presence of Lewis or Brønsted acid catalysts such as montmorillonite K10 clay [9], $\text{Ce}(\text{SO}_4)_2$ [10], iodine [11], $\text{K}_5\text{CoW}_{12}\text{O}_{40}\cdot 3\text{H}_2\text{O}$ [12], *p*-TSA [13], sulfamic acid [14], cation-exchanged resins [15], $\text{FeCl}_3\cdot\text{SiO}_2$ [16], silica-sulfuric acid [17], Brønsted acidic ionic liquid [18], $\text{Fe}(\text{HSO}_4)_3$ [19], $\text{Sr}(\text{OTf})_2$ [20], PPA-SiO₂ [21], oxalic acid [22], molybdophosphoric acid [23], ZrOCl_2 [24], and thiamine hydrochloride [25].

However, most of the synthetic protocols for 1-amidoalkyl-2-naphthol reported so far suffer from drawbacks such as unsatisfactory yields, long reaction times, and the production of environmental pollutants. Therefore, a great demand still exists for versatile, simple, and environmentally friendly processes whereby 1-amidoalkyl-2-naphthols may be formed under simple and practical conditions.

Herein, we report a convenient, mild and efficient procedure for a one-pot three-component synthesis of new 1-amidoalkyl 2-naphthol derivatives from various aryl and

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Scheme 1.

alkyl aldehydes, β -naphthol and different amides in the presence of DPA [26] as an effective and cost efficient catalyst under solvent-free conditions (Scheme 1).

2. Results and discussion

Initially, to optimize and find the best conditions, the reaction of β -naphthol (1 mmol), 4-nitrobenzaldehyde

(1 mmol) as the test substrate and benzamide (1.2 mmol) in the presence of DPA (0.1 mmol) was performed in different solvents, such as H_2O , EtOH/ H_2O (1:1), toluene, acetonitrile, and ethyl acetate (Table 1, entries 2–6) under reflux conditions. The reaction did not proceed at all in any of the solvents except in toluene in which the desired product was obtained in 48% yield. Then the model reaction was investigated under solvent-free conditions in

Table 1
Investigated conditions for preparation of 1-amidoalkyl-2-naphthol using of dodecylphosphonic acid (DPA) as catalyst.

Entry	Solvent	Catalyst (mol%)	Temp. ($^\circ\text{C}$)	Time (min)	Yield (%) ^a
1	Solvent-free	DPA (10 mol%)	90	20	88
2	H_2O	DPA (10 mol%)	Reflux	120	–
3	EtOH: H_2O	DPA (10 mol%)	Reflux	120	–
4	Toluene	DPA (10 mol%)	Reflux	120	48
5	Acetonitrile	DPA (10 mol%)	Reflux	120	–
6	Ethyl acetate	DPA (10 mol%)	Reflux	120	–
7	Solvent-free	DPA (5 mol%)	90	20	45
8	Solvent-free	DPA (20 mol%)	90	20	90
9	Solvent-free	DPA (10 mol%)	50	20	45
10	Solvent-free	DPA (10 mol%)	100	20	85

^a Isolated yields.

Table 2
The reaction of β -naphthol, amides and aldehydes in solvent-free conditions.

Entry	R^1	R^2	Product	Time (min)	Yield ^a (%)	Mp (lit. ($^\circ\text{C}$))
1	4- $\text{NO}_2\text{C}_6\text{H}_4$	C_6H_5	4a	20	88	240 (239–241 [22])
2	3- $\text{NO}_2\text{C}_6\text{H}_4$	C_6H_5	4b	20	90	241 (240–242 [27])
3	C_6H_5	C_6H_5	4c	20	90	235 (235–237 [24])
4	4- $\text{CH}_3\text{OC}_6\text{H}_4$	C_6H_5	4d	20	85	196 (197–199 [23])
5	4- $(\text{CH}_3)_2\text{CHC}_6\text{H}_4$	C_6H_5	4e	20	90	210
6	2- BrC_6H_4	C_6H_5	4f	20	88	229 (228–230 [14])
7	$\text{C}_6\text{H}_5\text{CH}_2\text{CH}_2$	C_6H_5	4g	30	80	158
8	$\text{C}_6\text{H}_5\text{CH}=\text{CH}$	C_6H_5	4h	30	85	234
9	9-Phenanthryl	C_6H_5	4i	20	81	251
10	1-Pyrenyl	C_6H_5	4j	20	86	246
11	2-Naphtyl	C_6H_5	4k	20	90	218
12	$\text{CH}_3(\text{CH}_2)_{10}$	C_6H_5	4l	30	70	166
13	CH_3CH_2	C_6H_5	4m	30	60	226
14	9-Anthryl	C_6H_5	4n	60	N.R	–
15	C_6H_5	CH_3	4o	20	88	230 (228–230 [11])
16	$\text{C}_6\text{H}_5\text{CH}=\text{CH}$	CH_3	4p	20	81	173 (174–175.5 [23])
17	9-Phenanthryl	CH_3	4q	20	86	273
18	2- BrC_6H_4	CH_3	4r	20	88	204
19	$\text{CH}_3(\text{CH}_2)_{10}$	CH_3	4s	30	72	110

^a Isolated yields.

the presence of DPA (10 mol%) at 90 °C and the desired product was formed in 88% yield (Table 1, entry 1). Decreasing of the yield was observed when the reaction was repeated in the presence of 5 mol% of the catalyst (Table 1, entry 7). Enhancement of the catalyst loading to 20 mol% did not show any effect on the yield or the reaction time (Table 1, entry 8).

Also the effect of temperature was studied and the results indicated that the best yield was provided at 90 °C (Table 1, entries 1, 9 and 10). At lower temperatures than that, the reaction did not proceed properly and most of the initial substances remained unreacted.

These optimized reaction conditions were then applied on the preparations of 1-amidoalkyl-2-naphthols with a variety of aromatic and aliphatic aldehydes (Table 2). Aromatic aldehydes bearing electron-donating and withdrawing substituents led to good yields of the desired 1-amidoalkyl-2-naphthols (Table 2, entries 1–6). Even hindered polynuclear aromatic aldehydes such as 2-naphthaldehyde, phenantherene-9-carbaldehyde and pyrene-1-carbaldehyde gave good yields under the same condition (Table 2, entries 9–11). In contrast to the previously reported methods in literature [11,19,23], in which aliphatic aldehydes were not transformed to the corresponding 1-amidoalkyl-2-naphthols, also in our study they were converted to the desired product in good yields when mixed with β -naphthol and benzamide in the presence of DPA under solvent-free conditions at 90 °C (Table 2, entries 12 and 13).

Using acetamide as an aliphatic amide, instead of benzamide, resulted in the corresponding 1-amidoalkyl-2-naphthols in good yields when it was treated with β -naphthol and aromatic or aliphatic aldehydes in the presence of DPA at 90 °C (Table 2, entries 15–19).

For showing efficiency of DPA compared to the reported catalysts in literature, including Lewis acids and Brønsted acids, a study was designed on the reaction of β -naphthol, acetamide, and benzaldehyde as a model. These data, which are shown in Table 3, revealed that DPA is a better catalyst than most of the conventional catalysts mentioned, with the exception being the protocols that

were aided by microwave or ultrasound (Table 3, entry 2 and 10).

3. Conclusion

In summary, we have therefore established a catalytic, environmental friendly route for the one-pot preparation of a range of new 1-amidoalkyl-2-naphthols with aromatic and aliphatic aldehydes by using DPA, which is commercially available and easy to produce, that has not been reported before. In addition, this method offers several significant advantages such as: high conversions, easy handling, short reaction times, easy and green work up, cost efficient and versatility. These advantages, in general, highlight this protocol as a useful and attractive methodology, among the methods reported in the literature, for the rapid synthesis of 1-amidoalkyl-2-naphthols as precursors of biological active 1-aminoalkyl-2-naphthols.

4. Experimental

The ^1H and ^{13}C NMR spectra were recorded on Bruker Advance DPX FT spectrometer at 250 and 62.9 MHz, respectively, and with TMS as an internal standard. Elemental analyses were performed on Thermofinnigan 1112 flash EA. The synthesized 1-amidoalkyl-2-naphthols were fully characterized with ^1H -NMR, ^{13}C -NMR spectroscopy and CHN analysis.

4.1. General procedure for the synthesis of 1-amidoalkyl-2-naphthols with dodecylphosphonic acid (DPA)

A mixture of β -naphthol (1 mmol), aromatic aldehyde (1 mmol) and amide (1.2 mmol) and DPA (0.1 mmol) in 5 mL round bottomed flask, which was equipped with a condenser, was heated at 90 °C. After completion of the reaction (monitored by TLC), the resulted powder was dissolved in 20 mL of hot ethanol. Then 20 mL of water was added to the reaction mixture. The resulting precipitate was filtered. Then the solid product was purified by recrystallization in aqueous ethanol.

Table 3

A comparison among efficiency of dodecylphosphonic acid (DPA) and some reported acids on the reaction of β -naphthol, benzaldehyde and acetamide.

Entry	Catalyst	Mole %	Time/yield (%) / condition
1	$\text{Fe}(\text{HSO}_4)_3$	5	65 min/83/solvent-free (85 °C) [19]
2	$\text{Fe}(\text{HSO}_4)_3$	5	7 min/90/solvent-free/microwave [19]
3	$\text{FeCl}_3 \cdot \text{SiO}_2$	0.025 g	11 min/86/solvent-free (120 °C) [16]
4	$\text{K}_9\text{CoW}_{12}\text{O}_{40} \cdot 3\text{H}_2\text{O}$	1	2 h/90/solvent-free (125 °C) [12]
5	K-10 clay	0.1 g	1.5 h/89/solvent-free (125 °C) [9]
6	I_2	5	4.5 h/87/solvent-free (125 °C) [11]
7	Molybdophosphoric Acid	0.12 g	3.5 h/94/ethylacetate (65 °C) [23]
8	PPA- SiO_2	0.03 g	5 min/80/solvent-free (120 °C) [21]
9	PTSA	10	15 h/89/ $\text{ClCH}_2\text{CH}_2\text{Cl}$ (rt) [13]
10	Sulfamic acid	50	15 min/89/solvent-free/sonication [14]
11	Sulfamic acid	50	4 h/72/solvent-free (28–30 °C) [14]
12	[TEBSA][HSO_4]	5	10 min/81/solvent-free (120 °C) [18]
13	ZrOCl_2	10	11 h/79/ $\text{ClCH}_2\text{CH}_2\text{Cl}$ (rt) [24]
14	Thiamine.HCl	10	4 h/88/EtOH [25]
15	Cyanuric chloride (TCT)	10	50 min/93/solvent-free (100 °C)
16	DPA	10	20 min/88/solvent-free (90 °C)

4.2. Spectral data for new compounds

N-((2-Hydroxynaphthalen-1-yl) (4-isopropylphenyl) methyl)benzamide (4e, C₂₇H₂₅NO₂): white solid, m.p 210 °C, ¹H NMR (250 MHz, DMSO-*d*₆): δ = 1.10 (d, 6H), 2.72 (m, 1H), 7.00 (d, 1H), 7.09 (m, 6H), 7.42 (m, 4H), 7.75 (m, 4H), 8.08 (d, 1H), 9.01 (d, 1H), 10.34 (s, 1H). ¹³C NMR (62 MHz, DMSO-*d*₆): δ = 23.81, 32.97, 49.11, 118.34, 122.61, 126.06, 126.49, 126.69, 127.02, 127.39, 128.13, 128.30, 128.45, 128.55, 129.20, 131.14, 131.34, 132.25, 134.30, 139.28, 146.64, 153.05, 165.50. Anal. Calcd: C, 82.00; H, 6.37; N, 3.54. Found: C, 81.89; H, 6.28; N, 3.48.

N-(1-(2-Hydroxynaphthalen-1-yl)-3-phenylpropyl) benzamide (4g, C₂₆H₂₃NO₂): white solid, m.p 158 °C, ¹H NMR (250 MHz, DMSO-*d*₆): δ = 2.04 (m, 2H), 2.74 (t, 2H), 5.98 (d, 1H), 7.13–7.24 (m, 6H), 7.40–7.47 (m, 4H), 7.66–7.82 (m, 4H), 8.02 (d, 1H), 8.66 (d, 1H), 10.16 (s, 1H). ¹³C NMR (62 MHz, DMSO-*d*₆): δ = 32.46, 35.60, 46.70, 118.59, 119.62, 122.38, 125.66, 126.22, 126.96, 127.33, 128.18, 128.26, 128.35, 128.45, 128.52, 131.13, 132.02, 134.63, 141.68, 152.90, 165.55. Anal. Calcd: C, 81.86; H, 6.08; N, 3.67. Found: C, 81.86; H, 5.98; N, 3.52.

(E)-N-(1-(2-Hydroxynaphthalen-1-yl)-3-phenylallyl) benzamide (4h, C₂₆H₂₃NO₂): white solid, m.p 234 °C, ¹H NMR (250 MHz, DMSO-*d*₆): δ = 7.08 (1H, d), 7.17 (d, 1H), 7.21 (m, 4H), 7.41 (m, 3H), 7.75 (m, 3H), 8.07 (d, 1H), 9.01 (d, 1H), 10.43 (s, 1H). ¹³C NMR (62 MHz, DMSO-*d*₆): δ = 48.15, 123.51, 127.61, 128.30, 131.27, 132.47, 133.60, 135.81, 137.28, 141.84, 158.24, 165.54. Anal. Calcd: C, 82.30; H, 5.58; N, 3.69. Found: C, 82.27; H, 5.55; N, 3.66.

N-((2-Hydroxynaphthalen-1-yl)(phenanthren-9-yl)methyl) benzamide (4i, C₃₂H₂₃NO₂): Pink solid, m.p 251 °C, ¹H NMR (250 MHz, DMSO-*d*₆): δ = 7.06 (m, 7H), 7.59 (m, 6H), 7.80 (m, 6H), 8.31 (d, 1H), 8.75 (d, 1H), 8.88 (s, 1H), 9.27 (d, 1H), 10.19 (s, 1H). ¹³C NMR (62 MHz, DMSO-*d*₆): δ = 48.15, 117.47, 118.80, 122.46, 122.62, 123.53, 124, 126.53, 126.61, 126.89, 127.07, 127.44, 128.19, 128.48, 128.63, 129.69, 130.41, 130.71, 131.23, 134.18, 153.83, 165.37. Anal. Calcd: C, 84.74; H, 5.11; N, 3.09. Found: C, 84.68; H, 5.05; N, 3.04.

N-((2-Hydroxynaphthalen-1-yl)(pyren-1-yl)methyl) benzamide (4j, C₃₄H₂₃NO₂): Yellow solid, m.p 246 °C, ¹H NMR (250 MHz, DMSO-*d*₆): δ = 7.22 (d, 1H), 7.25–7.46 (m, 5H), 7.80–8.49 (m, 14H), 8.85 (d, 1H), 9.37 (d, 1H), 10.33 (s, 1H). ¹³C NMR (62 MHz, DMSO-*d*₆): δ = 48.29, 117.96, 122.51, 122.93, 124.46, 125.20, 125.34, 126.18, 127.10, 127.22, 127.40, 127.76, 128.25, 128.50, 130.07, 130.19, 130.68, 134.15, 134.81, 153.68, 165.41. Anal. Calcd: C, 85.51; H, 4.85; N, 2.93. Found: C, 85.49; H, 4.81; N, 2.89.

N-((2-Hydroxynaphthalen-1-yl)(naphthalen-1-yl)methyl) benzamide (4k, C₂₈H₂₁NO₂): Pink solid, m.p 218 °C, ¹H NMR (250 MHz, DMSO-*d*₆): δ = 7.30 (d, 1H), 7.41–7.48 (m, 7H), 7.76–7.80 (m, 7H), 7.88 (d, 1H), 8.14 (d, 1H), 9.12 (d, 1H), 10.47 (s, 1H). ¹³C NMR (62 MHz, DMSO-*d*₆): δ = 49.44, 118.14, 118.66, 122.70, 124.37, 125.25, 125.64, 126.09, 127.15, 127.30, 127.64, 127.80, 128.38, 128.48, 128.63, 129.52, 131.45, 131.89, 132.67, 139.52, 153.28, 165.90. Anal. Calcd: C, 83.35; H, 5.25; N, 3.47. Found: C, 83.32; H, 5.19; N, 3.44.

N-(1-(2-Hydroxynaphthalen-1-yl)dodecyl)benzamide (4l, C₂₉H₃₇NO₂): white solid, m.p 167 °C, ¹H NMR (250 MHz, DMSO-*d*₆): δ = 0.88 (t, 3H), 1.23 (m, 18H), 1.67 (m, 2H), 6.02 (t, 1H), 7.21 (d, 1H), 7.36 (t, 1H), 7.53 (t, 3H), 7.65 (d, 1H), 7.83 (d, 2H), 8.23 (d, 1H), 8.64 (d, 1H), 10.18 (s, 1H). ¹³C NMR (62 MHz, DMSO-*d*₆): δ = 13.86, 22, 26.22, 28.87, 31.19, 33.76, 46.79, 118.54, 119.78, 122.33, 126.20, 126.84, 128.33, 131.06, 132.07, 134.63, 152.81, 165.29. Anal. Calcd: C, 80.70; H, 8.64; N, 3.25. Found: C, 80.74; H, 8.58; N, 3.24.

N-(1-(2-Hydroxynaphthalen-1-yl)propyl)benzamide (4m, C₂₀H₁₉NO₂): white solid, m.p 226 °C, ¹H NMR (250 MHz, DMSO-*d*₆): δ = 0.90 (t, 3H), 1.89 (m, 2H), 5.85 (t, 1H), 7.09 (d, 1H), 7.29 (dd, 1H), 7.43 (m, 4H), 7.66 (d, 1H), 7.75 (m, 3H), 8.18 (d, 1H), 8.61 (d, 1H), 10.08 (s, 1H). ¹³C NMR (62 MHz, DMSO-*d*₆): δ = 11.35, 26.91, 48.49, 118.57, 119.53, 122.34, 126.22, 126.87, 128.19, 131.06, 132.23, 134.71, 152.85, 165.37. Anal. Calcd: C, 78.66; H, 6.27; N, 4.59. Found: C, 78.65; H, 6.20; N, 4.55.

(E)- N-(1-(2-Hydroxynaphthalen-1-yl)-3-phenylallyl) acetamide (4p, C₂₁H₁₉NO₂): white solid, m.p 173 °C, ¹H NMR (250 MHz, DMSO-*d*₆): δ = 1.89 (s, 3H), 6.48 (d, 1H), 6.52 (m, 2H), 7.16 (m, 7H), 7.43 (m, 1H), 7.70 (m, 1H), 7.76 (m, 1H), 8.10 (d, 1H), 8.35 (d, 1H), 10.02 (s, 1H). ¹³C NMR (62 MHz, DMSO-*d*₆): δ = 27.87, 52.53, 123.51, 127.61, 128.30, 131.27, 132.47, 133.60, 135.81, 137.28, 141.84, 158.24, 173.93. Anal. Calcd: C, 79.47; H, 6.03; N, 4.41. Found: C, 79.38; H, 5.98; N, 4.33.

N-(2-Hydroxynaphthalen-1-yl)(phenanthren-9-yl) methyl) acetamide (4q, C₂₇H₂₁NO₂): white solid, m.p 273 °C, ¹H NMR (250 MHz, DMSO-*d*₆): δ = 1.89 (s, 3H), 7.08 (m, 7H), 7.59 (m, 6H), 7.80 (m, 6H), 8.31 (d, 1H), 8.78 (d, 1H), 8.87 (s, 1H), 9.27 (d, 1H), 10.20 (s, 1H). ¹³C NMR (62 MHz, DMSO-*d*₆): δ = 28.87, 48.18, 117.47, 118.80, 122.46, 122.62, 123.53, 124, 126.53, 126.61, 126.89, 127.07, 127.44, 128.19, 128.48, 128.63, 129.69, 130.41, 130.71, 131.23, 134.18, 154.83, 173.17. Anal. Calcd: C, 82.84; H, 5.41; N, 3.58. Found: C, 82.78; H, 5.34; N, 3.42.

N-((2-Bromophenyl)(2-hydroxynaphthalen-1-yl)methyl)acetamide (4r, C₁₉H₁₆BrNO₂): white solid, m.p 204 °C, ¹H NMR (250 MHz, DMSO-*d*₆): δ = 1.87 (s, 3H), 6.91 (d, 1H), 7.06–7.29 (m, 5H), 7.47 (t, 2H), 7.69 (m, 2H), 7.92 (d, 1H), 8.54 (d, 1H), 9.73 (s, 1H). ¹³C NMR (62 MHz, DMSO-*d*₆): δ = 22.28, 49.84, 116.95, 118.57, 122.18, 122.69, 122.78, 126.21, 126.82, 128.20, 128.42, 128.50, 129.31, 130.15, 132.49, 132.89, 141.26, 153.59, 168.42. Anal. Calcd: C, 61.64; H, 4.36; N, 3.78. Found: C, 61.58; H, 4.28; N, 3.74.

N-(1-(2-Hydroxynaphthalen-1-yl)dodecyl)acetamide (4s, C₂₄H₃₅NO₂): white solid, m.p 110 °C, ¹H NMR (250 MHz, DMSO-*d*₆): δ = 0.79 (t, 3H), 1.15 (m, 18H), 1.80 (s, 3H), 1.91 (m, 2H), 5.68 (t, 1H), 7.11 (d, 1H), 7.20 (m, 1H), 7.37 (m, 1H), 7.62 (d, 1H), 7.72 (d, 1H), 7.98 (d, 1H), 8.00 (d, 1H), 9.82 (s, 1H). ¹³C NMR (62 MHz, DMSO-*d*₆): δ = 13.87, 22.02, 22.67, 26.23, 28.62, 28.77, 28.90, 31.22, 33.56, 45.73, 118.49, 119.74, 122.13, 122.47, 125.98, 128.14, 128.24, 128.41, 132.23, 152.92, 168.36. Anal. Calcd: C, 78.00; H, 9.55; N, 3.79. Found: C, 77.94; H, 9.48; N, 3.75.

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