

Zn(OAc)₂·2H₂O-CATALYZED GREEN SYNTHESIS OF SUBSTITUTED 1-AMIDO/THIOAMIDOALKYL-2-NAPHTHOLS**

Pradeep Ramesh Rao TOTAWAR,^a Ravi VARALA^{b,*} and Jitendra S. PULLE^c

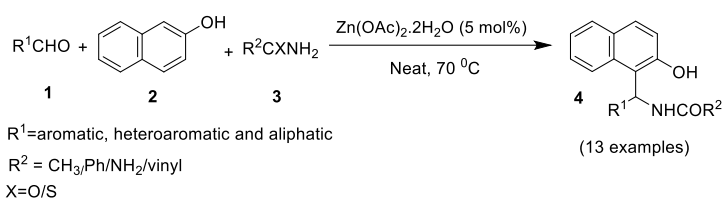
^aNagnath Arts, Commerce and Science College, Aundha Nagnath, Hingoli-431705, Maharashtra, India

^bScrips Pharma, Hyderabad-500 076, Telangana, India

^cShri Guru Buddhi Swami Mahavidyalaya, Purna, Parbhani-431511, Maharashtra, India

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Zn(OAc)₂·2H₂O (5 mol%)-catalyzed one-pot multi-component reaction of β-naphthol, aldehydes and amide/thioamides to produce corresponding amido-benzyl-naphthols in shorter reaction times (20 min–2 h) under neat conditions at 70 °C, is reported in good to excellent yields (58–95%).



INTRODUCTION

A number of pharmaceutical and agricultural agents have naphthalene framework. Some of the commonly known examples include LY326315 (a selective estrogen receptor I modulator, naproxen and nabumetone (non-steroidal anti-inflammatory drugs), naphazoline and pronethalol (cardiovascular agents), 1-naphthaleneacetic acid and 2-naphthoxyacetic acid (plant growth regulators), terbinafine & naftifine (antifungal agents) etc. (Figure 1).¹ In literature, 2-naphthol and 2-naphthol derivatives are also reported as bactericides and antioxidants.

Multi-component reactions (MCRs), which have the advantages of selectivity, superior yields, faster reaction times, atom economy, and environmentally friendly nature, are essential tools in the toolbox of synthetic and medicinal chemists for the creation of a wide variety of new chemical entities (NCEs).^{2–5} Due to its numerous applications, the synthesis of new heterocyclic compounds of medical significance has attracted a lot of attention in recent years. The synthesis of nitrogen heterocycles with an amidobenzyl-naphthols moiety is crucial among a wide range of heterocyclic molecules.

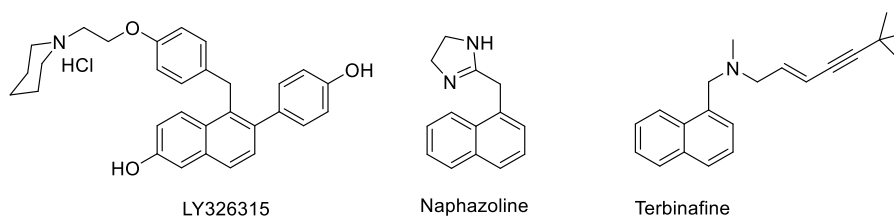


Fig. 1 – Representative naphthalene derivatives of biological relevance.

* Corresponding author: ravivarala@gmail.com; +91-9618286529

**Supplementary information on <https://www.icf.ro/rrch/> or <https://revroum.lew.ro>

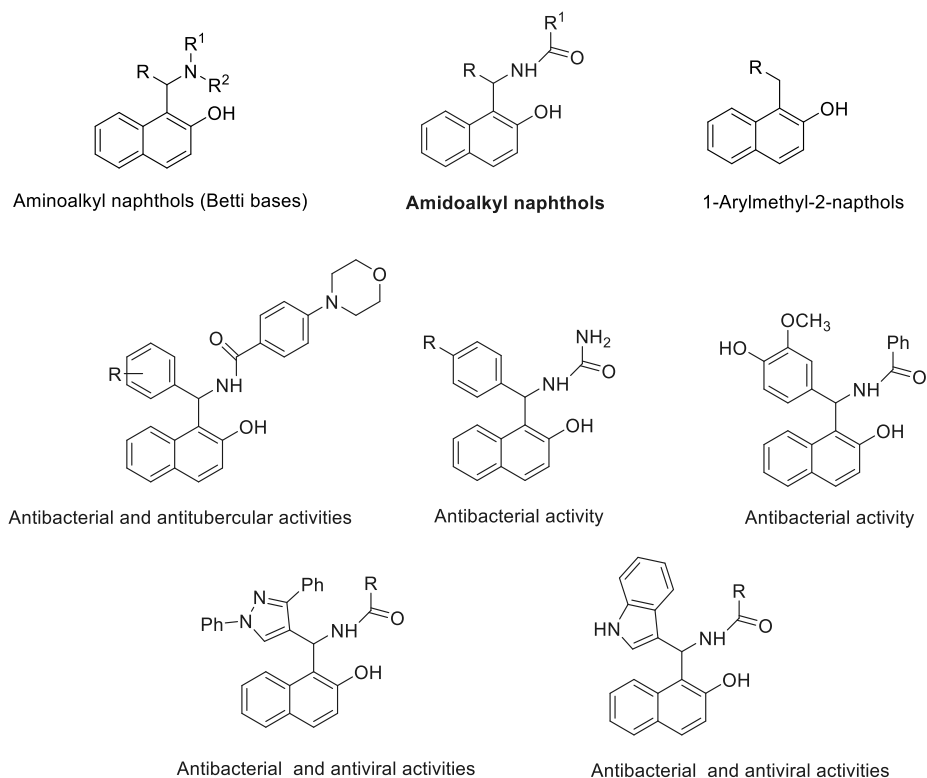


Fig. 2 – The structure of some biologically active amidoalkyl naphthols.

Numerous nucleosides, antibiotics, and human immunodeficiency virus protease inhibitors, including lopinavir and ritonavir, are among the powerful medications that belong to the class of amidoalkyl naphthols (Betti bases) and amidoalkyl naphthols with 1,3-amino-oxygenated functional group (Figure 2).⁶ A modified Mannich process for condensation of β -naphthol, aldehydes, and ammonia or with other amines is typically used in the traditional synthesis of Betti bases.⁷

A Ritter reaction is the nucleophilic addition of a nitrile to a carbenium ion (alcohols as precursors) under the influence of an acid, followed by the hydrolysis to produce the corresponding amide.⁸ A three-component Ritter-type reaction using β -naphthol, different aldehydes, and amides to create 1-amidoalkyl-2-naphthols is one of the most significant in organic chemistry. Because of their medicinal and biological properties, amidoalkyl naphthols have been studied in synthetic and medicinal organic chemistry.⁹ It is known that *ortho*-quinone methides are produced when β -naphthol reacts with aromatic aldehydes in the presence of an acid catalyst (*O*-QMs). The identical *O*-QMs that were produced *in situ* have been used to create 1-amidoalkyl 2-naphthol derivatives by reacting with acetamide or benzamide. The precursors for the production of 1,3-oxazine and/or 1,3-oxazinone

derivatives are 1-amidoalkyl 2-naphthols. Amidoalkyl naphthols have also been transformed into bioactive aminoalkyl naphthol derivatives using amide hydrolysis. Figure 2 shows the structures of a few 1-amidoalkyl-2-naphthols having biological characteristics.

Various catalysts such as sulfamic acid, $\text{Sr}(\text{OTf})_2$, $\text{NaHSO}_4 \cdot \text{SiO}_2$, ionic liquids, $\text{H}_4\text{SiW}_{12}\text{O}_{40}$, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, $\text{P}_2\text{O}_5/\text{SiO}_2$, H_3BO_3 , *p*-TSA, $\text{RuCl}_2(\text{PPh}_3)_3$, trichloroacetic acid or cobalt (II) chloride, $\text{Mg}(\text{ClO}_4)_2$, anhyd. $\text{ZnCl}_2/\mu\text{w}$, $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$, Amberlite IR-120, hexachlorocyclotriphosphazene (HCCP), nano-graphene oxide, adipic acid, nano- SnO_2 , MgSO_4 , deep eutectic solvent ($[\text{cholineCl}][\text{ZnCl}_2]_3$, etc.¹⁰⁻⁴⁸ were employed for the purpose. Numerous of these methods call for high temperatures, readily accessible and expensive chemicals, large volumes of dangerous organic solvents, and other factors. Therefore, investigating the novel catalytic system is still necessary today and presents a difficult problem to organic chemists. It is preferable to do so utilizing an easy synthetic process using less or no harmful solvents to address their limitations. With reference to other pertinent recent literature reports⁴⁹⁻⁵¹ as well as our prior successful results with $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$,⁵²⁻⁵⁵ we are disclosing our results for the synthesis of amidobenzyl naphthols here.

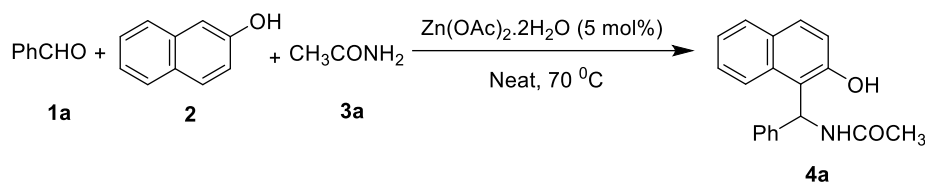
RESULTS AND DISCUSSION

The reaction of β -naphthol (1 mmol, **2**), benzaldehyde (1 mmol, **1a**), and acetamide (1.2 mmol, **3a**) was chosen as the control experiment with $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$ as the catalyst at 70 °C in order to optimize the reaction conditions (Scheme 1).

It was found that a slight excess of amide was beneficial. Initially, the reaction was carried out under varied solvent or solvent-free settings to optimize the reaction parameters (Table 1). The optimal conditions for the reaction in terms of reaction time (20 min) and yield (94%) were discovered to be solvent-free (entry 1, Table 1). Non-polar solvents had little effect on the reaction, however polar solvents produced a poor yield of product (**4a**). The control reaction was determined

to be viable at 70 °C, and temperature increases of 10 °C up to 90 °C were also examined. The related product was produced in minimal or small amounts when the temperature was gradually lowered till ambient temperature. The reaction can produce the best results at 5 mol%, according to studies of catalyst loading (5, 7.5, and 10 mol%). Higher catalyst concentrations (10 and 15 mol%) had no effect on the yield either up or down.

Later, various zinc catalysts were examined under controlled circumstances to determine how well they supported the control reaction (Table 2). $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$ was found to be the most effective catalyst when compared to the other catalysts tested (ZnCl_2 , zinc dust, ZnO , $\text{Zn}(\text{OTf})_2$, $\text{Zn}(\text{NO}_3)_2$, and ZnCO_3) in terms of reaction time and yield.



Scheme 1 – Control experiment for the synthesis of amidoalkyl naphthols.

Table 1

Effect of solvent

Entry	Solvent	Time (min)	Reaction condition	Yield (%) ^a
1 ^b	Neat	20	70 °C 80 °C 90 °C	94 94 92
2	THF	60	reflux	17
3	toluene	30	reflux	45
4	acetone	60	reflux	-
5	CH ₃ CN	60	reflux	27
6	DMF	60	70 °C	-

^aIsolated yields; ^b $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$ loading: (95%, 10 mol%); (94%, 7.5 mol%) and (94%, 5 mol%)

Table 2

Effect of zinc salts

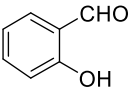
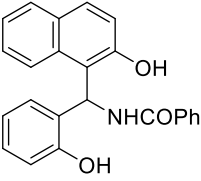
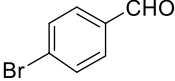
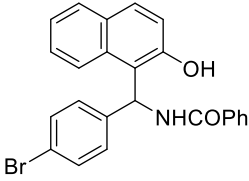
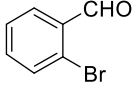
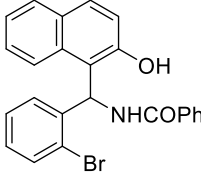
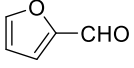
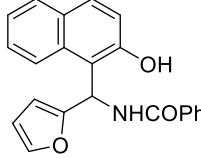
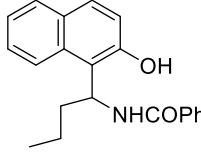
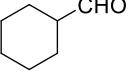
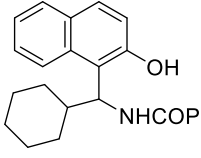
Entry ^b	Zinc salt	Yield ^a (%)
1	ZnCl_2	32
2	Zinc dust	21
3	ZnO	-
4	$\text{Zn}(\text{OTf})_2$	15
5	$\text{Zn}(\text{NO}_3)_2$	7
6	ZnCO_3	0
7	$\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$	94

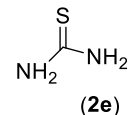
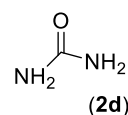
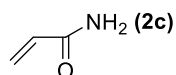
^aIsolated yields.

Table 3

Zn(OAc)₂•2H₂O-Catalyzed synthesis of amidobenzyl naphthols

Entry	Aldehyde	Amide	Product	Reaction time (min/h)	Yield(%) ^a
4a		2a		20	94 ¹⁵
4b		2b		30	95 ¹⁵
4c		2d		30	92 ¹⁵
4d		2e		30	84 ¹²
4e		2b		30	91 ¹⁵
4f		2d		20	90 ¹³
4g		2c		20	89 ²²
	CH ₃ CONH ₂ (2a)	PhCONH ₂ (2b)			

Entry	Aldehyde	Amide	Product	Reaction time (min)	Yield (%) ^a
4h		2b		45	78 ⁴⁶
4i		2b		20	91 ⁴⁴
4j		2b		45	82 ⁴⁷
4k		2b		120	58 ⁴⁶
4l	CH ₃ CH ₂ CH ₂ CHO	2b		120	0
4m		2b		240	0

^aYields refer to pure isolated products

The structure of the related product is confirmed by the presence of a doublet at 8.44 ppm in the ¹H NMR spectrum for the -CH-NH group together with other aromatic protons, a peak at 169.7 ppm for the C=O group in the ¹³C NMR, and a prominent peak at 1640 cm⁻¹ for the -NHCOCH₃ group in the IR. At 70 °C in each of these instances, the corresponding 1-amidoalkyl-2-naphthols were produced in good yields without

the development of any undesired byproducts, such as dibenzoxanthenes, which are typically seen when strong acids are present.

A variety of aldehyde and amide precursors were employed to react with 2-naphthol once the optimal conditions for the aforementioned reaction (5 mol% Zn(OAc)₂·2H₂O at 70 °C under neat circumstances) were discovered. With various amides/thioamides, benzaldehyde interacted to

produce the desired compounds in good to exceptional yields (entries 4a-d, Table 3). In high to outstanding yields (78–92%) and with a brief reaction time, a range of aromatic aldehydes with both electron-donating and electron-withdrawing groups were converted to amidoalkyl naphthols (entries 4e-j, Table 3) with short reaction times (20–45 min). By using IR and NMR spectrum analysis to identify all known products, the results were verified by contrasting them with those found in the literature. According to the findings in Table 3, it can be generally inferred that the reaction is being influenced by the type of substitution in the aromatic ring.

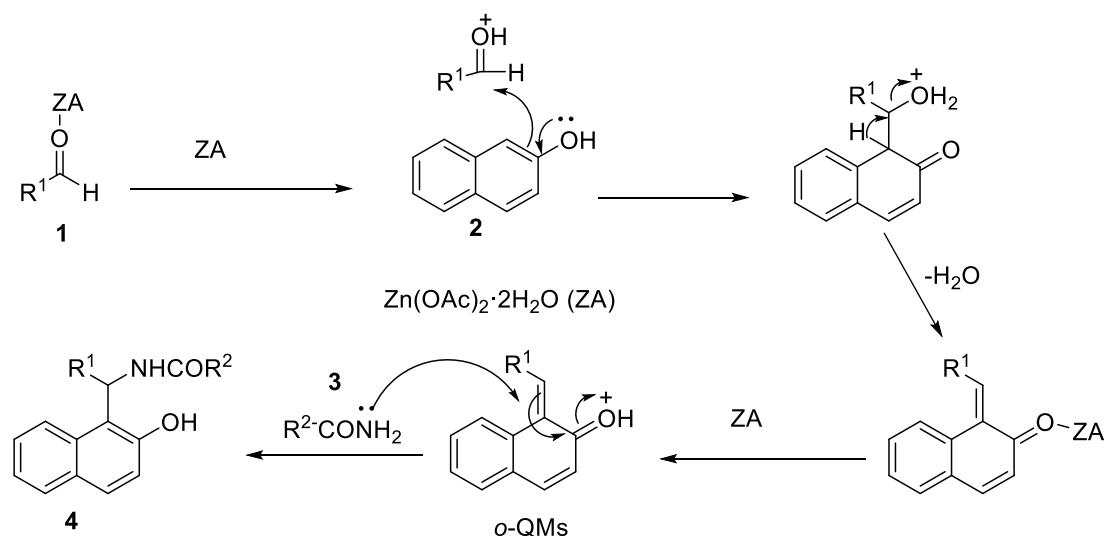
The reaction time was quicker with several of the aldehydes when electron withdrawing groups were utilized in para-positions as opposed to electron donating groups (entries 4f, 4g and 4i, Table 3). *Ortho* substituents reduce reaction yield, perhaps as a result of the steric effect (entries 4h and 4j, Table 3).

It is interesting to observe that the reaction with electron-withdrawing group aldehydes, like 4-nitro benzaldehyde was completed in less time than those with electron-donating group aldehydes (entries 4f and 4g, Table 3). According to earlier findings, the quicker rate of the current reaction may be due to the lower energy of LUMO of alkenes carrying electron-withdrawing groups (a carbonyl group, like in the intermediate *ortho*-quinone methides), as opposed to alkenes containing electron-donating groups. The type of substitution in amide had no impact on the reaction duration or yield either. Heterocyclic aldehydes,

such as furfuraldehyde, generated the corresponding product in a moderate yield (entry 4k, Table 3). Aliphatic aldehydes reacted slowly and produced side products, making it impossible to isolate the intended product (entries 4l and 4m, Table 3). Furthermore, the control reaction's results (92%) were replicated by the catalytic procedure at a greater scale (1 g), demonstrating the viability of amidoalkyl naphthol synthesis.

As described in the literature,⁴³ Scheme 2 illustrates a process for the synthesis of 1-amidoalkyl-2-naphthols (**4**). Initially $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$ coordinates to the oxygen atom of benzaldehyde, thereby, increasing the electrophilicity of aldehyde (**1**) and shortening the reaction time. The extremely reactive and transient intermediate *ortho*-quinone (*o*-QMs) is produced by condensation of β -naphthol (**2**) with the activated aldehyde (ZA). Amide (**3**) has been combined with the analogous *ortho*-quinone methides that were produced *in situ* to produce 1-amidoalkyl-2-naphthol derivatives.

As indicated by Table 4, this approach is preferable when compared to some of those described in the literature, in terms of both reaction durations and product yields. It is evident that the catalyst for this multicomponent reaction, $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$, is also as effective as or more effective (Table 4, entry 16). This approach does not involve any ionic liquids, dangerous solvents like chloroform, or specialized equipment like an ultrasound machine or a microwave, in contrast to some of the previously reported procedures.



Scheme 2 – Plausible mechanism for the formation of 1-amidoalkyl naphthol.

Table 4

Comparison of Zn(OAc)₂·2H₂O with other catalysts reported in the literature under solvent-free conditions for the control reaction as reference

Entry	Catalyst	Reaction conditions (°C)	Time (min)	Yield (%) ^a	Reference
1	Dodecylphosphonic acid (10 mol%)	90	20	88	33
2	H ₄ SiW ₁₂ O ₄₀ (5 mol%)	125	120	90	26
3	Sulfamic acid (50 mol%)	30, (((15	89	34
4	Iodine (5 mol%)	125	330	85	35
5	Fe(HSO ₄) ₃ (5 mol%)	85	65	83	36
6	Montmorillonite K-10 (0.1 g)	125	90	89	37
7	K ₅ CoW ₁₂ O ₄₀ ·3H ₂ O (1 mol%)	125	120	90	38
8	[TEBSA][HSO ₄] IL (5 mol%)	120	10	87	24
9	Silica sulfuric acid (0.02 g)	25	120	85	39
10	SiO ₂ ·HClO ₄ (0.6 mol%)	125	40	89	40
11	NaHSO ₄ ·SiO ₂ (6 mol%)	125	9	95	41
12	Cation-exchanged resins (0.25 g)	110	20	81	42
13	ZnO Nps (20 mol%)	120-130	30	87	43
14	Amberlite IR-120 (0.16 g)	360 W, microwave	5	94	30
15	Ba ₃ (PO ₄) ₂ nanoparticles (10 mo%)	100	45	87	20
16	Zn(OAc)₂·2H₂O	70	20	94	Present work

^aIsolated yields

EXPERIMENTAL

Typical procedure for the synthesis of 1-amidoalkyl-2-naphthols

The mixture of the aromatic aldehyde 1 (2 mmol), 2-naphthol (2 mmol), amide (2 mmol), and Zn(OAc)₂·2H₂O (5 mol%) was stirred at 70 °C in an oil bath for the appropriate time (Table 1, monitored by TLC, ethyl acetate:*n*-hexane = 1:3). Then, acetone (15 mL) was added and the reaction mixture was filtered. The solid catalyst was washed with acetone (2×10 mL) and dried under vacuum. Pure 1-amidoalkyl-2-naphthols were afforded by evaporation of the solvent followed by recrystallization from ethanol. All products were characterized by spectral data and compared with their physical data with the literature.

Representative spectra data:

N-[(2-Hydroxynaphthalen-1-yl)-phenylmethyl]acetamide (entry 4a, Table 1): White solid, mp= 227-229 °C. FT-IR (KBr, cm⁻¹): 3400, 3248, 3062, 1640, 1583, 1513, 1437, 1372, 1337, 1304, 1277, 1252, 1235, 1208, 1168, 1103, 1061, 1029, 987, 933, 877, 838, 807, 742, 697, 659, 625, 569. ¹H NMR (400 MHz, DMSO-*d*₆): δ 1.97 (s, 3H, CH₃), 7.11-7.16 (m, 4H, ArH), 7.20-7.27 (m, 4H, ArH), 7.35 (t, *J*=7.4 Hz, 1H, ArH), 7.75-7.81 (m, 3H, ArH and CH), 8.44 (d, *J*=8.4 Hz, 1H, NH), 9.98 (s, 1H, ArOH). ¹³C NMR (100 MHz, DMSO-*d*₆): δ 23.1, 48.3, 118.9, 119.3, 122.8, 126.5, 126.7, 128.4, 128.9, 129.0, 129.7, 132.8, 143.1, 153.6, 169.7.

N-((2-Hydroxynaphthalen-1-yl)(phenyl)methyl)benzamide (entry 4b, Table 1): White solid, mp = 234-236 °C. IR (KBr, cm⁻¹): 3418, 3061, 3021, 1629, 1570, 1435, 821, 751, 613. ¹H NMR (500 MHz, DMSO-*d*₆): δ 10.34 (s, 1H), 9.02 (d, *J* = 8.5 Hz, 1H), 8.09 (d, *J* = 8.5 Hz, 1H), 7.89-7.78 (m, 4H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.47 (dd, *J* = 15.0, 7.5 Hz, 3H), 7.34-7.23 (m, 7H), 7.22-7.17 (m, 1H). ¹³C NMR (125 MHz, DMSO-*d*₆): δ 166.2, 153.7, 142.5, 134.8, 132.8, 131.9, 129.9, 129.1, 129.0, 128.9, 128.7, 127.6, 127.2, 127.0, 126.9, 123.2, 119.2, 118.8, 49.7

N-((2-Hydroxy naphthalen-1-yl)-phenyl-methyl]-urea (entry 4c, Table 1): White solid, mp = 184-186 °C; ¹H NMR (200 MHz, DMSO-*d*₆): δ 10.28 (s, 1H), 7.85-7.15 (m, 12H), 6.90 (s, 2H), 5.70 (s, 2H); ¹³C NMR (50 MHz, DMSO-*d*₆): δ 159.3, 153.6, 144.3, 132.8, 131.1, 130.0, 129.4, 129.1, 128.9, 128.6, 128.4, 127.4, 123.3, 120.4, 119.2, 48.4; Mass (ES/MS): *m/z* 291 (M-H, 100%)

N-((2-Hydroxynaphthalen-1-yl)(*p*-tolyl)methyl)benzamide (entry 4e, Table 1): Light yellow solid. mp= 224-226 °C. FT-IR (KBr, cm⁻¹): 3410, 3153, 3062, 1632, 1575, 1538, 1515, 1487, 1436, 1413, 1344, 1278, 1145, 1074, 1025, 940, 876, 815, 751, 703. ¹H NMR (400 MHz, DMSO-*d*₆): δ 2.24 (s, 3H), 7.08 (d, *J*=7.1 Hz, 2H), 7.18 (d, *J*=7.1 Hz, 2H), 7.24-7.31 (m, 3H), 7.44-7.57 (m, 4H), 7.79-7.87 (m, 4H), 8.08 (d, *J*=8.0 Hz, 1H, NH), 9.01 (d, *J*=8.0 Hz, 2H), 10.33 (s, 1H, ArOH); ¹³C NMR (100 MHz, DMSO-*d*₆): δ 21.0, 49.5, 118.9, 119.2, 123.1, 126.9, 127.2, 127.6, 128.8, 129.0, 129.1, 129.2, 129.8, 131.9, 132.8, 134.9, 136.1, 139.5, 153.6, 166.1

N-((4-Nitrophenyl)-(2-hydroxy naphthalen-1-yl)methyl] urea (entry 4f, Table 1): Yellow solid. M.p.: 221-223 °C; IR (KBr, cm⁻¹) ν 3445, 3347, 3299, 3006, 2836, 1661, 1595, 1539, 1509, 1454, 1370, 1237, 999 cm⁻¹. ¹H NMR (300 MHz, DMSO-*d*₆): δ 10.26 (s, 1H), 7.97-7.73 (m, 3H), 7.62-7.23 (m, 7H), 7.05 (s, 2H) 6.58 (s, 2H) ppm. ¹³C NMR (90 MHz, DMSO-*d*₆): δ 160.1, 150.1, 134.4, 132.6, 129.5, 129.2, 128.1, 127.9, 127.7, 127.2, 125.7, 123.7, 122.5, 120.8, 119.9, 49.2 ppm.

N-((2-Hydroxynaphthalen-1-yl)(2-hydroxyphenyl)methyl) benzamide (entry 4h, Table 1): White solid, mp = 234-236 °C. IR (KBr, cm⁻¹) ν 3592, 3205, 2363, 1607, 1565, 1349, 816, 752, 687, 588. ¹H NMR (500 MHz, DMSO-*d*₆): δ 10.18 (s, 1H), 9.64 (s, 1H), 8.92 (s, 1H), 8.24 (d, *J* = 9.0 Hz, 1H), 7.84 (d, *J* = 7.0 Hz, 2H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.73 (d, *J* = 9.0 Hz, 1H), 7.53 (t, *J* = 7.0 Hz, 1H), 7.50-7.37 (m, 4H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.28 (t, *J* = 7.0 Hz, 1H), 7.20 (d, *J* = 9.0 Hz, 1H), 7.05 (t, *J* = 7.0 Hz, 1H), 6.80 (d, *J* = 7.5 Hz, 1H), 6.68 (t, *J* = 7.0 Hz, 1H). ¹³C NMR (125 MHz, DMSO-*d*₆): δ 165.6, 155.3, 153.6, 135.0, 133.0, 131.7, 129.5, 129.2, 128.9,

128.8, 128.5, 128.2, 127.6, 126.6, 123.8, 123.3, 122.9, 119.1, 119.3, 115.8, 46.2

N-(4-Bromophenyl)(2-hydroxynaphthalen-1-yl)methyl benzamide (entry 4i, Table 1): White solid, mp = 197-199 °C. IR (KBr, cm⁻¹): ν 3418, 3182, 1628, 1576, 1341, 810, 724, 585. ¹H NMR (500 MHz, DMSO-*d*₆): δ 10.36 (s, 1H), 9.02 (d, *J* = 8.5 Hz, 1H), 8.07 (d, *J* = 8.5 Hz, 1H), 7.89-7.84 (m, 3H), 7.82 (d, *J* = 9.0 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.53-7.44 (m, 5H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.30-7.21 (m, 4H). ¹³C NMR (125 MHz, DMSO-*d*₆): δ 166.4, 153.7, 142.0, 134.7, 132.7, 132.0, 131.5, 130.1, 129.2, 129.1, 129.0, 128.9, 127.7, 127.3, 123.2, 120.1, 119.1, 118.3, 49.3.

N-(Furan-2-yl)(2-hydroxynaphthalen-1-yl)methyl benzamide (entry 4k, Table 1): Brown solid, mp = 221-223 °C. IR (KBr, cm⁻¹): ν 3409, 2197, 1632, 1572, 1437, 816, 745, 597. ¹H NMR (500 MHz, DMSO-*d*₆): δ 10.21 (s, 1H), 9.09 (d, *J* = 8.0 Hz, 1H), 8.18 (d, *J* = 8.5 Hz, 1H), 7.89-7.77 (m, 4H), 7.59-7.52 (m, 2H), 7.47 (t, *J* = 7.5 Hz, 3H), 7.36-7.27 (m, 2H), 7.23 (d, *J* = 9.0 Hz, 1H), 6.36 (dd, *J* = 3.0, 2.0 Hz, 1H), 6.13 (d, *J* = 3.0 Hz, 1H). ¹³C NMR (125 MHz, DMSO-*d*₆): δ 166.1, 154.5, 153.9, 142.5, 142.5, 134.7, 132.7, 131.9, 130.0, 129.0, 128.9, 128.8, 128.7, 127.7, 127.0, 123.4, 123.0, 119.0, 116.8, 110.9, 110.9, 107.2, 107.2, 45.0.

CONCLUSION

The current method using Zn(OAc)₂·2H₂O provides an effective synthesis of 1-amidobenzyl naphthols by a one-pot three component coupling of aldehydes, amides, and 2-naphthol in moderate to excellent yields with the following noteworthy features: use of a widely accessible and affordable catalyst; tolerability of various functional groups; and clean reaction conditions. Also described are the limitations of Zn(OAc)₂·2H₂O catalysis.

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