An Efficient Solvent-Free Protocol for the Synthesis of 1-Amidoalkyl-2-naphthols using Silica-Supported Molybdotophosphoric Acid

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Abstract: A highly efficient, green and simple solvent-free method for the synthesis of 1-amidoalkyl-2-naphthols *via* one-pot multi-components condensation of 2-naphthol, aromatic aldehydes and amides in the presence of catalytic amount of silica-supported molybdotophosphoric acid ($\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O}/\text{SiO}_2$, 3.17 mol%) is described. The reactions proceed rapidly and the title compounds are produced in high to excellent yields.

Keywords: 1-Amidoalkyl-2-naphthol, Molybdotophosphoric acid, Heteropolyacid, Solvent-free, Multi-components reaction, 2-Naphthol.

Introduction

1-Amidoalkyl-2-naphthol derivatives are of importance because they can be easily converted to the important biologically active compounds, 1-aminoalkyl-2-naphthols, by amide hydrolysis reaction. The hypotensive and bradycardiac properties have been reported for this class of compounds^{1,2}. 1-Amidoalkyl-2-naphthols can be also converted to 1,3-oxazine derivatives³. 1,3-Oxazines have potentially different biological activities including antibiotic⁴, antitumor⁵, analgesic⁶, anticonvulsant⁷, antipsychotic⁸, antimalarial⁹, antianginal¹⁰, antihypertensive¹¹ and antirheumatic properties¹². One-pot multi-components condensation of 2-naphthol, aromatic aldehydes and amide derivatives or acetonitrile has been used as a practical synthetic routes toward 1-amidoalkyl-2-naphthols¹³⁻²². Several Lewis and Brønsted acids have been applied for this transformation including $\text{Ce}(\text{SO}_4)_2$ ¹³,

Montmorillonite K10¹⁴, iodine¹⁵, cation-exchanged resins¹⁶, NaHSO₄·H₂O¹⁷, Fe(HSO₄)₃¹⁸, sulfamic acid/ultrasound¹⁹, HClO₄/SiO₂²⁰, cyanuric chloride²¹ and K₃CoW₁₂O₄₀·3H₂O²². However, many of the reported methods suffer from one or more of the following drawbacks: (i) low product yield, (ii) prolonged reaction time, (iii) the use of large amount of reagent, (iv) the use of toxic reagents and (v) incompatibility with the green chemistry protocols. Therefore, search for finding a protocol for the synthesis of 1-aminoalkyl-2-naphthols that are not associated with the above disadvantages is still relevant.

Heteropolyacids are important catalysts in industries for fine organic synthetic processes related with fine chemicals such as synthesis of antioxidants, medicinal preparations, vitamins and biologically active substances²³. They are more active catalysts than conventional inorganic and organic acids for a variety of organic reactions²⁴⁻³¹. Among heteropolyacids, molybdatophosphoric acid (H₃PMo₁₂O₄₀·xH₂O) is a good promoter due to its high acid strengths, thermal stabilities, low reducibility and atom economy^{24,28-31}.

Recently, the use of catalysts and reagents supported on solid supports under solvent-free conditions has been developed because such reagents not only cause to simplify the purification processes but also help to prevent the releasing of toxic reaction residues into the environment³²⁻⁴². Moreover, due to moisture sensitivity of some reagents; application of them in organic reactions is difficult. This problem can be solved by supporting of them on solid supports³²⁻⁴². Handling of solid-supported reagents is also easy. Silica gel is one of the most interesting solid supports and is inexpensive, reusable, commercially available and environmentally benign which has been used in various organic transformations³⁶⁻⁴². Consequently, it is noteworthy that the combination of safe supported-catalysis with the use of solvent-free technique represents a suitable way toward the so-called “ideal synthesis”³²⁻⁴².

In this paper, we report a new, efficient and simple method for the synthesis of 1-aminoalkyl-2-naphthols from 2-naphthol, aromatic aldehydes and amides in the presence of H₃PMo₁₂O₄₀·xH₂O/SiO₂ as a green catalytic system under solvent-free conditions (Scheme 1). It is worth noting that this method has none of the above-mentioned disadvantages at all.

Experimental

All chemicals were purchased from Merck or Fluka Chemical Companies. Silica gel 60, 0.063-0.200 mm (7-230 mesh ASTM) was applied as support. The structures of known compounds were identified by comparison of their melting points and spectral data with those in the authentic samples. The ¹H NMR (250 or 300 MHz) and ¹³C NMR (62.5 or 75 MHz) were run on a Bruker Avance DPX-250 FT-NMR spectrometer (δ in ppm). Microanalyses were performed on a Perkin-Elmer 240-B microanalyzer. Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes.

Procedure for the preparation of H₃PMo₁₂O₄₀·xH₂O/SiO₂ (33% w/w) catalytic system

A mixture of H₃PMo₁₂O₄₀·xH₂O (1 g) and SiO₂ (2 g) was ground vigorously to give H₃PMo₁₂O₄₀·xH₂O/SiO₂ catalytic system as a yellow powder (3 g).

General procedure for the synthesis of 1-amidoalkyl-2-naphthol derivatives

To a well-ground mixture of 2-naphthol (2 mmol, 0.288 g), amide (2.4 mmol) and H₃PMo₁₂O₄₀·xH₂O/SiO₂ (0.350 g, 3.17 mol%) in a 10 mL round-bottomed flask connected to a reflux condenser, was added aldehyde (2 mmol) and the resulting mixture was stirred in an oil-bath (120 °C) for the appropriate time. After completion of the reaction, as monitored by TLC, the reaction mixture was cooled to room temperature, and was added to a column chromatography on silica gel and eluted with EtOAc/*n*-hexane (1/2) to give pure product.

General procedure for the condensation of 2-naphthol with terephthalaldehyde and acetamide leading to 1-amidoalkyl-2-naphthol and bis-1-amidoalkyl-2-naphthol

A well-ground mixture of 2-naphthol (4 mmol, 0.576 g), acetamide (4.8 mmol, 0.284 g), silica-supported $\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O}$ (0.700 g, 6.34 mol%) and terephthalaldehyde (2 mmol, 0.268 g) in a 10 mL round-bottomed flask connected to a reflux condenser was stirred in an oil-bath (120 °C) for 30 min. Afterward, the reaction mixture was cooled to room temperature and warm aqueous ethanol (15%, 20 mL) was added to it and stirred for 5 min (1-amidoalkyl-2-naphthol is soluble in warm aqueous ethanol, however, bis-1-amidoalkyl-2-naphthol is insoluble in this solvent). During this time, the crude 1-amidoalkyl-2-naphthol was dissolved in the ethanol and pure bis-1-amidoalkyl-2-naphthol was remained; thus, two products were easily separated by filtration. Then, the crude 1-amidoalkyl-2-naphthol was purified by column chromatography on silica gel eluted with EtOAc/*n*-hexane (1/2).

Selected spectral data of the products

N-[(2-Hydroxynaphthalen-1-yl)(phenyl)methyl]acetamide (I)

White solid; m.p. 239-240 °C (lit.¹³ 241-243 °C); ¹H NMR (DMSO-*d*₆, 250 MHz): δ 2.06 (s, 3H), 7.01-7.20 (m, 9H), 7.65-7.73 (m, 3H), 8.11 (d, *J* = 7.7 Hz, 1H), 9.69 (s, 1H); ¹³C NMR (DMSO-*d*₆, 62.5 MHz): δ 23.5, 41.3, 118.8, 120.2, 122.0, 123.9, 124.9, 125.7, 127.6, 128.1, 128.3, 128.5, 128.6, 134.2, 144.0, 152.6, 169.6; Anal. calcd. for C₁₉H₁₇NO₂: C, 78.33; H, 5.88; N, 4.81. Found: C, 78.54; H, 6.01; N, 4.65.

N-[(2-Hydroxynaphthalen-1-yl)(3-nitrophenyl)methyl]acetamide (8)

Pale yellow solid; m.p. 238-240 °C (lit.¹⁶ 236-237 °C); ¹H NMR (DMSO-*d*₆, 250 MHz): δ 2.06 (s, 3H), 7.15-7.49 (m, 6H), 7.78-8.04 (m, 5H), 8.54 (d, *J* = 8.1 Hz, 1H), 10.12 (s, 1H); ¹³C NMR (DMSO-*d*₆, 62.5 MHz): δ 23.3, 48.1, 118.1, 118.7, 120.5, 122.3, 123.8, 125.7, 127.3, 128.4, 129.1, 129.6, 130.8, 133.1, 134.1, 144.5, 148.7, 152.9, 169.8; Anal. calcd. for C₁₉H₁₆N₂O₄: C, 67.85; H, 4.79; N, 8.33. Found: C, 68.04; H, 4.68; N, 8.47.

N-[(2-Hydroxynaphthalen-1-yl)(4-chlorophenyl)methyl]acetamide (10)

Pale yellow solid; m.p. 220-222 °C (lit.¹³ 224-227 °C); ¹H NMR (DMSO-*d*₆, 250 MHz): δ 2.07 (s, 3H), 7.06 (m, 2H), 7.14-7.24 (m, 5H), 7.68-7.77 (m, 3H), 7.98 (d, *J* = 7.4 Hz, 1H), 8.16 (d, *J* = 7.1 Hz, 1H), 9.90 (s, 1H); ¹³C NMR (DMSO-*d*₆, 62.5 MHz): δ 23.2, 47.6, 118.8, 119.8, 122.1, 123.7, 125.8, 126.9, 127.4, 128.3, 128.6, 129.3, 129.9, 134.0, 143.4, 152.5, 169.8; Anal. calcd. for C₁₉H₁₆ClNO₂: C, 70.05; H, 4.95; N, 4.30. Found: C, 69.78; H, 4.79; N, 4.42.

N-[(4-Chloro-phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl]nicotinamide (16)

White solid; m.p. 206-209 °C; ¹H NMR (DMSO-*d*₆, 300 MHz): δ 7.27-7.50 (m, 9H), 7.83 (d, *J* = 8.7 Hz, 2H), 8.10 (d, *J* = 8.4 Hz, 1H), 8.27 (d, *J* = 7.8 Hz, 1H), 8.71 (d, *J* = 4.4 Hz, 1H), 9.12 (s, 1H), 9.35 (d, *J* = 7.6 Hz, 1H), 10.38 (s, 1H); ¹³C NMR (DMSO-*d*₆, 75 MHz): δ 49.4, 118.2, 119.1, 123.1, 123.5, 123.9, 127.2, 128.6, 128.9, 129.2, 130.1, 130.4, 131.7, 132.8, 135.8, 141.2, 149.1, 152.4, 153.9, 165.4; MS *m/z*: 388.3 (M⁺); Anal. calcd. for C₂₃H₁₇ClN₂O₂: C, 71.04; H, 4.41; N, 7.20. Found: C, 71.32; H, 4.28; N, 7.36.

N-[(4-Formylphenyl)-(2-hydroxynaphthalen-1-yl)methyl]acetamide (17)

Pale yellow solid; m.p. 237-240 °C; ¹H NMR (DMSO-*d*₆, 300 MHz): δ 2.02 (s, 3H), 6.91-7.39 (m, 6H), 7.79-7.82 (m, 4H), 8.41 (d, *J* = 6.6 Hz, 1H), 8.56 (d, *J* = 7.6 Hz, 1H), 9.93 (s, 1H), 10.10 (s, 1H); ¹³C NMR (DMSO-*d*₆, 75 MHz): δ 23.0, 48.4, 118.6, 118.8, 122.9, 123.5, 126.3, 127.1, 128.9, 129.1, 129.9, 130.1, 132.7, 134.9, 150.6, 153.7, 170.1, 193.1; MS *m/z*: 319.2 (M⁺); Anal. calcd. for C₂₀H₁₇NO₃: C, 75.22; H, 5.37; N, 4.39. Found: C, 75.01; H, 5.25; N, 4.53.

N-[4-[Acetylamino-(2-hydroxy-naphthalen-1-yl)-methyl]-phenyl]-(2-hydroxy-naphthalen-1-yl)-methyl]acetamide (**18**)

White solid; m.p. 277-279 °C; ¹H NMR (DMSO-*d*₆, 300 MHz): δ 2.01 (s, 6H), 7.17-7.39 (m, 12H), 7.77-7.93 (m, 6H), 8.53 (d, *J* = 8.2 Hz, 2H), 10.09 (s, 2H); ¹³C NMR (DMSO-*d*₆, 75 MHz): δ 23.1, 48.2, 118.9, 119.2, 122.8, 126.3, 126.8, 128.9, 129.0, 129.6, 132.7, 140.8, 140.9, 153.6, 169.7; MS *m/z*: 504.5 (M⁺); Anal. calcd. for C₃₂H₂₈N₂O₄: C, 76.17; H, 5.59; N, 5.55. Found: C, 75.86; H, 5.43; N, 5.73.

Results and Discussion

To optimize the reaction conditions, at first we selected the synthesis of *N*-((2-hydroxynaphthalen-1-yl)(phenyl)methyl)acetamide (**1**) as a model reaction (Scheme 1); thus, to a well-ground mixture of 2-naphthol (2 mmol), acetamide (2.4 mmol) and molybdatophosphoric acid (0.117 g, 3.17 mol%) was added benzaldehyde (2 mmol) and the resulting mixture was stirred in an oil-bath (120 °C) under solvent-free conditions.

Scheme 1

However, in these conditions, a highly sticky reaction mixture was obtained and the product was obtained in 79% yield after 15 min. Increasing the reaction time or the temperature didn't improve the yield. The synthesis of 1-amidoalkyl-2-naphthol **1** was also examined in different solvents including DMF, DMSO, NMP, HMPTA, THF, EtOH and EtOH/H₂O (1/1, v/v); but, low yields (34-65%) of the product were obtained in long reaction times (2-4 h) in the solution conditions. The yield increased to 91% when the reaction was carried out in the presence of silica-supported H₃PMo₁₂O₄₀·xH₂O (33% w/w) as catalyst under solvent-free conditions (Table 1, entry 3). To choose suitable amount of H₃PMo₁₂O₄₀·xH₂O/SiO₂ as well as reaction temperature, the multi-components condensation of 2-naphthol (2 mmol), benzaldehyde (2 mmol) and acetamide (2.4 mmol) was tested using different amount of the catalytic system at range of 100-130 °C in the absence of solvent. The results are summarized in Table 1. As it can be seen in Table 1, the reasonable results were obtained in the presence of 0.35 g (3.17 mol%) of H₃PMo₁₂O₄₀·xH₂O/SiO₂ at 120 °C.

Table 1. The condensation of 2-naphthol (2 mmol), benzaldehyde (2 mmol) and acetamide (2.4 mmol) using different amount of H₃PMo₁₂O₄₀·xH₂O/SiO₂ (33% w/w) at range of 100-130 °C

Entry	Catalyst Amount, g	Temperature, °C	Time, min	Yield ^a , %
1	0.25 (2.28 mol%)	120	30	68
2	0.30 (2.73 mol%)	120	20	85
3	0.35 (3.17 mol%)	120	15	91
4 ^b	0.40 (3.65 mol%)	120	15	91
5	0.35 (3.17 mol%)	100	40	60
6	0.35 (3.17 mol%)	110	25	77
7	0.35 (3.17 mol%)	130	15	90

^aIsolated yield. ^bIncreasing the amount of H₃PMo₁₂O₄₀·xH₂O/SiO₂ didn't improve the reaction results.

To assess generality and scope of the method, 2-naphthol was reacted with various aromatic aldehydes and amides. The results are displayed in Table 2. As it is shown in Table 2, using this method, all aldehydes including benzaldehyde as well as aromatic aldehydes possessing electron-withdrawing substituents, electron-releasing substituents or halogens on their aromatic rings afforded the desired 1-amidoalkyl-2-naphthols in high to excellent yields and in short reaction times (Table 2, compounds **1-13**). The method was also successfully worked when benzamide or nicotinamide was used instead of acetamide in the reaction (Table 2, compounds **14-16**).

Table 2. The Solvent-free synthesis of 1-aminoalkyl-2-naphthols from 2-naphthol, aromatic aldehydes and amides using $\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O}/\text{SiO}_2$ at 120 °C

Compound Number	Product	Time, min	Yield ^a , %	M.p. °C (Lit.)
1		15	91	239-240 (241-243) ¹³
2		20	87	223-225 (222-223) ¹⁷
3		25	83	198-200 (199-202) ¹⁷
4		23	88	183-185 (184-186) ¹³
5		23	90	200-202 (201-204) ¹⁷
6		35	82	231-233 (235-236) ¹³
7		15	90	247-249 (248-250) ¹⁷
8		15	91	238-240 (236-237) ¹⁶
9		20	86	184-186 (180-182) ¹³

Contd...

10		15	86	220-222 (224-227) ¹³
11		20	80	196-198 (194-196) ¹⁹
12		15	85	226-228 (227-229) ¹⁷
13		15	87	206-208 (203-205) ¹⁶
14		60	82	237-239 (234-236) ¹⁶
15		60	84	214-216 (216-217) ¹⁶
16		60	75	206-209

^aIsolated yield.

The condensation of 2-naphthol with bis-aldehydes (terephthalaldehyde) and acetamide was also rapidly performed using $\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O}/\text{SiO}_2$ (Scheme 2). When 1 equivalent of 2-naphthol and 1 equivalent of the aldehyde were reacted with 1.2 equivalents of acetamide using $\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O}/\text{SiO}_2$ (3.17 mol%); after 20 min, 1-amidoalkyl-2-naphthol **17** and bis-1-amidoalkyl-2-naphthol **18** were obtained in 46 and 19%, respectively. However, the use of 2 equivalents of 2-naphthol, 1 equivalent of terephthalaldehyde and 2.4 equivalents of acetamide in the presence of $\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O}/\text{SiO}_2$ (6.34 mol%) gave compounds **17** and **18** in 16 and 75%, correspondingly, within 30 min.

Scheme 2

To compare the applicability and the efficiency of silica-supported $\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O}$ with the reported catalysts for the synthesis of 1-amidoalkyl-2-naphthols, we have tabulated turn-over frequency {TOF = yield (%) / [reaction time (min) $\times$ mol% of catalyst]} of these catalysts to perform the condensation of 2-naphthol with benzaldehyde and acetamide in Table 3. As Table 3 indicates, $\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O} / \text{SiO}_2$ is superior to the previously reported catalysts in term of TOF.

Table 3. Comparison of the results of the condensation of 2-naphthol with benzaldehyde and acetamide catalyzed by $\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O} / \text{SiO}_2$ with those obtained by the recently reported catalysts.

Catalyst	Catalyst, mol%	Time, min	Yield, %	TOF ^a , min ⁻¹	Ref.
$\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O} / \text{SiO}_2$	3.17	15	91	1.91	- ^b
$\text{Ce}(\text{SO}_4)_2$	100	2160	72	0.00033	13
Montmorillonite K10	0.1 g	90	89	-	14
I_2	5	330	85	0.051	15
$\text{Fe}(\text{HSO}_4)_3$	5	65	83	0.255	18
Sulfamic acid	51.5	15	89	0.115	19
Cyanuric chloride	10	10	91	0.91	21
$\text{K}_5\text{CoW}_{12}\text{O}_{40} \cdot 3\text{H}_2\text{O}$	1	120	90	0.75	22

^aTurn-over frequency. ^bOur catalyst.

Conclusion

In summary, we have developed a new protocol for the synthesis of 1-aminoalkyl-2-naphthols as biologically interesting compounds *via* the one-pot multi-components condensation of 2-naphthol, aromatic aldehydes and amides using silica-supported $\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O}$ under solvent-free conditions. The advantages of this method are efficiency, generality, high yield, relatively short reaction time, cleaner reaction profile, simplicity, ease of preparation of the catalytic system, ease of product isolation, and compliance with the green chemistry protocols.

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