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COPPER NITRATE TRIHYDRATE CATALYZED EFFICIENT ONE POT SYNTHESIS OF AMIDOALKYLNAPHTHOL DERIVATIVES.

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ABSTRACT:

In the present study we extend the scope of the $Cu(NO_3)_2.3H_2O$ catalyzed synthesis of amido alkyl naphthols and the results are presented here. In order to optimize the reaction conditions initially we studied the efficacy of $Cu(NO_3)_2.3H_2O$ by taking catalytic amount of 10 mol% and benzaldehyde (1 mmol) β-naphthol (1 mmol) and acetamide (1.2 mmol) in acetonitrile (10 ml) as model reaction.We have developed a practical and new, general efficient procedure for the one pot synthesis of amidoalkyl naphthols by coupling of various aromatic aldehydes with amides urea and β-naphthol using $Cu(NO_3)_2.3H_2O$ as catalyst. The present protocol has several advantages of readily available, inexpensive catalyst, mild reaction conditions, easy handling, excellent yields, greater selectivity, operational and experimental simplicity. We believe that $Cu(NO_3)_2.3H_2O$ catalyzed methodology will definitely be a valuable addition to the existing process in the field of amidoalkyl naphthols.

Keywords: *Amidoalkyl naphthols, β-naphthol, Multi component reaction, $Cu(NO_3)_2.3H_2O$.*

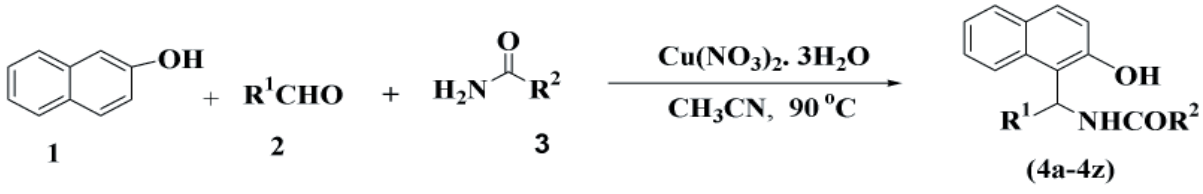
INTRODUCTION

Multicomponent reactions (MCRs) have been proved to be a very elegant and rapid way to access complex structures in a single synthetic operation from simple building blocks, and show high atom-economy, high selectivity and procedural simplicity due to the formation of carbon-carbon and carbon-heteroatom bonds in one-pot and generally afford good yields.[1] Multi-component reactions have attracted considerable attention in organic synthesis as they can produce target products in a single operation without isolating the intermediates and thus reducing the reaction times and energy input.[2, 3]

Compounds bearing 1,3-amino-oxygenated functional motifs are common in a variety of biologically important natural products and potent drugs, including a number of nucleoside antibiotics and HIV protease inhibitors, such as ritonavir and lipinavir.[4] Moreover, 1-amidoalkyl naphthol can be easily hydrolyzed to 1-aminoalkylnaphthol, which shows biological activities like hypotensive, bradycardiac effect and catalyst in asymmetric synthesis.[5,6]

The importance of amidoalkyl naphthols for their synthesis has attracted renewed attention and various improved procedures have been reported. Several alternative and efficient methods have been developed for the synthesis of amidoalkylnaphthol derivatives by multi component reaction of β-naphthol, aldehyde and amide in the presence of different catalysts such as Montmorillonite K10clay,[7] $Ce(SO_4)_2$,[8] $K_5CoW_{12}O_{40}.3H_2O$,[9] p-TSA,[10] Sulfamic acid/ultrasound,[11] Ionic liquids,[12] Indion-130,[13] $Al(H_2PO_4)_3$,[14] $Fe(HSO_4)_3$,[15] $Yb(OTf)_3$,[16] Wetcyanuric chloride,[17] Al_2O_3-$HClO_4$,[18] Silica chloride (SiO_2-Cl)/ultrasound,[19] indium(III)chloride,[20] $Sr(OTf)_2$,[21] P_2O_5,[22] $NaHSO_4.SiO_2$,[23] $H_4SiW_{12}O_{40}$,[24] Zeolite H-BEA,[25] N,N,N',N'-Tetrabromobenzene-1,3-disulfonamide(TBBD),[26] $KHSO_4$,[27] tritylchloride[28] Bismuth(III) nitrate pentahydrate,[29] H_3BO_3,[30] $Mg(ClO_4)_2$,[31] MNPs–SO_3H,[32] and Succinic acid.[33] In which some of often involves the use of expensive reagents, hazard solvents and tedious workup.

$Cu(NO_3)_2.3H_2O$ has been extensively used as a mild catalyst for a variety of organic trans formations.[34,35b,37-40] In continuation of our work to develop new organic transformations,[34-37] we would like to report a highly efficient route for the synthesis of amidoalkylnaphthol derivatives by one pot multi component-coupling of β-naphthol, aldehydes, amides/urea catalyzed by commercially available, inexpensive, mild $Cu(NO_3)_2.3H_2O$ as catalyst in good to excellent yields. (Scheme-1)



$R^2 = CH_3, C_6H_5, NH_2$

Scheme-1

Experimental

MATERIALS AND METHODS

Chemicals were purchased from Merck and Fluka and directly used for the synthesis. Thin layer chromatography (TLC): precoated silica gel plates (60 F254, 0.2mm layer; E. Merck). ^{1}HNMR (Avance 300 MHz) spectra were recorded in DMSO using TMS as internal standard. Melting points (m.p.) were determined on a Fischer-Johns melting point apparatus. IR and MS were recorded on a Thermo Nicolet Nexus 670 FT-IR Spectrometer and Finnegan MAT 1020 Mass spectrometer operating at 70 eV.

General procedure for the synthesis of amidoalkylnaphthol derivatives**:** A mixture of β-naphthol (1 mmol) aldehyde (1mmol) amides (1.2 mmol) or urea (1.2 mmol) and $Cu(NO_3)_2.3H_2O$ (10mol%) in acetonitrile (10 ml) was refluxed at 90°C for the time specified in (Table-3) after completion of the reaction as indicated by TLC, the reaction mixture was cooled to room temperature and washed with water and extracted with ethyl acetate, dried over Na_2SO_4 concentrated under vacuum and the crude mixture was purified by recrystalization from ethanol to afford pure product.

Spectral data of the selected compounds:

N-[phenyl-(2-hydroxy-naphthalen-1-yl) methyl]acetamide **(4a):** IR(KBr): 3406, 3250, 2920, 1640, 1583, 1519, 1437,1369, 1338, 1278, 1275, 1190, 1029, 987, 940, 875, 840, 810, 743 cm^{-1}; 1HNMR (300 MHz, DMSO-d6)δ = 9.98 (s, 1H), 8.45–8.42 (d, 1H), 7.84–7.74(m, 3H), 7.37–7.11 (m, 9H), 1.97 (s, 3H) ppm; MS(EI): m/z(%) 292 (M+).

N-[(4-Methyl phenyl)-(2-hydroxy-naphthalen-1-yl)methyl]acetamide **(4b):** IR (KBr): 3420, 3320, 3058, 1621, 1580, 1561, 1520, 1466, 1392, 1298, 1202, 1145, 1051, 945, 890, 748, 745, 719 cm^{-1}; ^{1}HNMR (300 MHz, DMSO-d_6): δ = 1.96 (s, 3H), 2.21 (s, 3H), 7.08-7.03 (m, 5H), 7.19 (d, J = 8.8 Hz, 1H), 7.24 (t, J = 7.1 Hz, 1H), 7.34 (m, 1H), 7.74 (d, J = 8.8 Hz, 1H), 7.78 (d, J = 7.9 Hz, 1H), 7.82 (br, 1H), 8.36 (d, J =8.1 Hz, 1H), 9.91 (s,1H) ppm; MS(EI): m/z (%) 305 (M$^+$).

N-[(4-Methoxy phenyl)-(2-hydroxy-naphthalen-1-yl)methyl]acetamide **(4c):** IR(KBr): 3396, 3078, 3002, 2967, 2832, 2787, 2704, 2614, 1627, 1581, 1515, 1438, 1378, 1334, 1304, 1279, 1268, 1179, 1088, 1075, 1043, 983, 930, 880, 850, 822, 814, 8035, 745 cm^{-1}; ^{1}HNMR (300 MHz, DMSO-d6) δ= 10 (s, 1H), 8.4 (d, 1H), 7.80–7.73 (m, 4H), 7.35 –7.04 (m, 6H), 2.5 (s, 3H), 1.953 (s, 3H) ppm; MS(EI) m/z: 322 (M$^+$).

N-[4-Nitro phenyl)-(2-hydroxy-naphthalen-1-yl)methyl]acetamide **(4i)**: IR(KBr): 3391, 3072, 1640, 1602, 1524, 1439, 1352, 1281, 1246, 1167, 1066, 1093, 983, 935, 883, 855, 825, 734, 750 cm–1; 1HNMR (300 MHz, DMSO-d6) δ= 10.11 (s, 1H), 8.59–8.56 (d, 1H), 8.14 –8.12 (m, 2H), 7.83–7.79 (m, 3H), 7.41 –7.38 (m, 3H), 7.28 – 7.16 (m, 2H), 2.02 (s, 3H) ppm; MS(EI) m/z: 337 (M$^+$).

N-[2-Hydroxy naphthalen-1-yl)-(naphthalen-1-yl)methyl]acetamide **(4m):** IR(KBr): 3420 3300, 2316, 1659, 1640, 1449, 1523, 748 Cm^{-1}; ^{1}HNMR (300 MHz, DMSO-d_6): δ= 9.41 (s,1H, OH), 8.52 (d, J = 8.5 Hz, 1H), 8.12 (d, J = 8.1 Hz, 1H), 7.81–7.85(m, 7H, Ar–H), 7.28 –7.34 (m, 6H, Ar–H), 2.88 (s, 3H, CH_3) ppm; MS(EI) m/z: 341(M$^+$).

N-[3-Nitro phenyl)-(2-hydroxy-naphthalen-1-yl)methyl]benzamide **(4u):** IR (KBr): 3369, 3278, 3099, 2978, 2956, 1640, 1578,1543, 1522, 1521, 1480, 1439, 1347, 1308, 1280, 1207, 1171, 1093, 1070, 963, 934, 867, 820, 740 cm–1; 1H NMR (300 MHz, DMSO-d6) δ 10.42 (s, 1H), 9.15–9.13 (d, 1H), 8.11–8.08 (m, 4H), 7.90– 7.82 (m, 5H), 7.73 –7.71 (m, 2H), 7.61–7.38 (m, 6H) ppm; MS(EI) m/z: 398 (M$^+$).

N-[4-Nitro phenyl)- (2-hydroxy-naphthalen-1-yl)methyl]urea **(4w) :** IR(KBr): 3481, 3405, 3377, 3179, 3062, 2924, 2849, 1716, 1655, 1600, 1517, 1439, 1346, 1257, 1140, 1109, 1018, 853, 825, 746 cm–1; 1H NMR (300 MHz, DMSO-d6) δ 9.957 (s, 1H), 7.82–7.73 (m, 3H), 7.41–7.27 (m, 1H), 7.24 –7.11 (m, 3H), 6.89(m, 2H), 6.75 – 6.72 (m, 3H), 5.82 (s, 2H), 3.65 (s, 3H) ppm; MS(EI): m/z (%) 322(M^+).

RESULTS AND DISCUSSION

In the present study we extend the scope of the $Cu(NO_3)_2.3H_2O$ catalyzed synthesis of amido alkylnaphthol derivatives and the results are presented here. In order to optimize the reaction conditions initially we studied the efficiency of $Cu(NO_3)_2.3H_2O$ by taking catalytic amount of 10 % mol and benzaldehyde (1 mmol) β-naphthol (1 mmol) and acetamide (1.2 mmol) in acetonitrile (10 ml) as model reaction, the reaction gave the corresponding *N*-[phenyl-(2-hydroxynaphthalen-1-yl) methyl]-acetamide with 92% yield in 4hours, with refluxing at 90°C (Table-3). In the absence of $Cu(NO_3)_2.3H_2O$ even up to 6 h no reaction was observed although the amount of catalyst has been optimized to 10% mol, (5% mol) also worked when longer reaction times were employed, while screening of various catalysts we found that $Cu(NO_3)_2.3H_2O$ was more effective than other nitrates tested in terms of isolated yields (92%) (10 Table-1), we choose $Cu(NO_3)_2.3H_2O$ as the suitable catalyst for further reactions due to its easy availability, cost effectiveness, easy handling intrigued by these observations we have then tested the efficiancy of several copper salts such as $Cu(OAc)_2$, Cu (Cl, Br, I) along with $Cu(NO_3)_2.3H_2O$ on the model reaction and among the copper salts screened $Cu(NO_3)_2.3H_2O$ was found to be best both in terms of reaction time and yields. The model reaction was performed in various solvents using $Cu(NO_3)_2.3H_2O$ as the catalyst to identify the best condhtions for the reaction. A range of solvents such as $CHCl_3$, DCM, THF, DMSO, toluene, H_2O and acetonitrile were examined and acetonitrile emerged as the solvent of choice in terms of reaction kinetics and product yields (Table-2). Encouraged by the results obtained for benzaldehyde we generalized the reaction scope for a number of other aromatic functionalities chloro, fluoro, methyl, methoxy nitro were tolerated and gave good yields.

Table- 1: Screening of various catalysts (metal nitrates) (10% mol) on model reaction between β -naphthol (1 mmol) benzaldehyde (1mmol), acetamide (1.2 mmol), in acetonitrile (10 ml) heating at 90 °C .

Entry	Catalyst	Time (h)	Yield (%)a
1	Ce(NH4)2.(NO3)6	6	45
2	Ni(NO3)3.6H2O	5.5	50
3	Al(NO3)3.9H2O	6	50
5	Zn(NO3)2.6H2O	5	60
6	AgNO3	5	50
7	Fe(NO3)3.9H2O	5.5	60
8	Rh(NO3)3.2H2O	5	76
9	La(NO3)3.6H2O	5.5	65
10	Cu(NO3)2.3H2O	4	92

a = Isolated yields

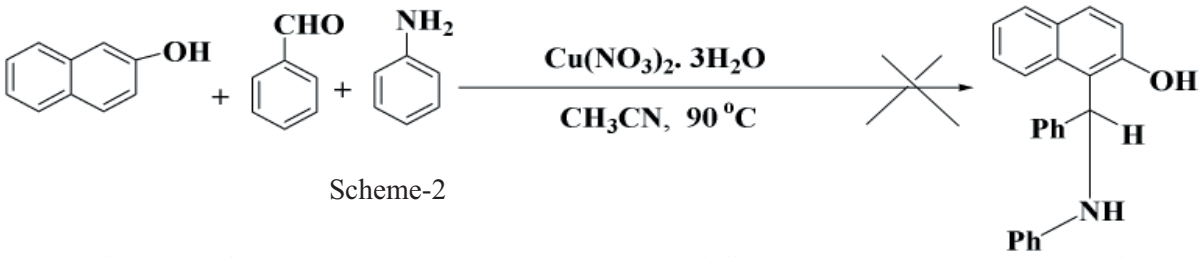
Table- 2: Screening of various solvents (10ml) on model reaction between β-naphthol (1 mmol) benzaldehyde (1mmol), acetamide (1.2 mmol), in the presence of $Cu(NO_3)_2.3H_2O$ (10 mol %) heating at 90°C.

Entry	Solvent	Time (h)	Yield (%)a
1	DCM	5.5	40
2	DMF	6	60
3	THF	6	55
4	H2O	7	40
5	CH3CN	4	92
6	EtOAc	5	54
7	CHCl3	5	70
8	DEA	5	68
9	Ethanol	5.5	55

a = Isolated yields

Aliphatic aldehydes such as butyraldehyde, propanaldehyde (Table-3) the reaction gave the corresponding amidoalkynaphthol derivatves in

good yield. In case of hereoaromatic aldehyde we have isolated the product with impurity (Table-3). Aromatic aldehydes carrying either electron-withdrawing or electron-donating groups reacted successfully and gave the desired product in high yields. A possible mechanism for this transformation is proposed in the literature,[7,10, 19, 29, 32] reaction of β-naphthol with aldehydes in the presence of lewis acid catalyst is known to give *ortho*-quinone methides (*o*-QMs). The (*o*-QMs) generated insitu, have been reacted with amides via conjugate addition to form 1-amidoalkyl-2-naphthol derivatives (Table-3). On the other hand, the reaction with amine such as aniline was utilized and no amino alkyl naphthols were obtained. (Scheme-2)



Scheme-2

Table-3 Synthesis of amidoalkyl naphthols in the presence of Cupric Nitrate Trihydrate (10mol%)

Entry	R^1	R^2	Product [a]	Time (h)[c]	Yield [b] (%)	m.p.	[Ref]
1	C_6H_5	CH_3	4a	4	92	242-244	32
2	4-$CH_3C_6H_4$	CH_3	4b	4	90	222-223	32
3	4-$OCH_3C_6H_4$	CH_3	4c	4	90	183-184	32
4	2,4 -$Cl_2C_6H_3$	CH_3	4d	4.5	85	202-204	32
5	4-ClC_6H_4	CH_3	4e	4.5	89	237-239	33
6	4 -$N(CH_3)_2C_6H_4$	CH_3	4f	4.5	82	120-123	32
7	C_6H_4N	CH_3	4g	4.5	63	193-194	31
8	2 -HOC_6H_4	CH_3	4h	4	80	216-218	31
9	4 -$NO_2C_6H_4$	CH_3	4i	4	92	251-253	33
10	C_6H_4-CH=CH	CH_3	4j	4.5	75	185-188	31
11	2 -ClC_6H_4	CH_3	4k	4.5	80	213-214	32
12	4-FC_6H_4	CH_3	4l	4.5	90	229-231	32
13	$C_{10}H_7$	CH_3	4m	4.5	80	228-230	32
14	3-OH4-$CH_3OC_6H_3$	CH_3	4n	4	83	237-239	32
15	CH_3-CH_2-CH_2	CH_3	4o	5	78	224-227	32
16	CH_3-CH_2	CH_3	4p	5	75	178-180	32
17	C_6H_5	C_6H_5	4q	4	92	238-240	32
18	4 -$NO_2C_6H_4$	C_6H_5	4r	4	90	240-242	33
19	4-$CH_3OC_6H_4$	C_6H_5	4s	4	90	209-212	32
20	2 -ClC_6H_4	C_6H_5	4t	4.5	85	286-288	32
21	3-$NO_2C_6H_4$	C_6H_5	4u	4.5	89	233-235	33
22	C_6H_5	NH_2	4v	4	82	179-181	32
23	4 -$NO_2C_6H_4$	NH_2	4w	4	79	163-165	33
24	3-$CH_3OC_6H_4$	NH_2	4x	4	80	243-245	33
25	3-$NO_2C_6H_4$	NH_2	4y	4	92	191-193	33
26	4-$CH_3C_6H_4$	NH_2	4z	4.5	88	115-118	33

a = All the products are characterized by spectral analysis, b = Isolated yields , c = reflux at 90°

CONCLUSION:

We have developed a practical and new, general efficient procedure for the one pot synthesis of amidoalkylnaphthol derivatives by coupling of various aromatic aldehydes with amides urea and β-naphthol using $Cu(NO_3)_2.3H_2O$ as catalyst. The present protocol has several advantages of readily available, inexpensive catalyst, mild reaction conditions, easy handling, excellent yields, greater selectivity, operational and experimental simplicity. We believe that $Cu(NO_3)_2.3H_2O$ catalyzed methodology will definitely be a valuable addition to the existing process in the field of amidoalkylnaphthol derivatives.

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