ISSN PRINT 2319 1775 Online 2320 7876

 UGC CARE Listed ( Group -I) Journal Volume 10, Iss 09, 2021

# AQUOUS ZINC TETRAFLUROBORATE SOLUTION CATALYSED SOLVENT FREE SYNTHESIS OF 2-AMIDOALKYL NAPHTHOL

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A complex molecule is synthesized in two or more stepwise procedure. This often involves isolation and purification of intermediates and alteration of reaction conditions for the next step. Now, many research groups have aimed for the realization of the concept of ideal synthesis by the development of multi-step, single operation processes for the construction of complex molecules. Here several bonds are formed in one step without isolating the intermediates which commonly known as Multi-component reactions (MCRs).

Multi-component reactions (MCRs) exhibit a high atom economy and selectivity. In contrast to the multi-step syntheses the MCRs need minimal work, and they have often quantitative yields and fewer byproducts. MCRs offer molecular diversity and complexity in a fast and often experimentally simple fashion.[1,2] For this reason, MCRs are particularly well suited for diversity oriented synthesis[3,4] and library synthesis of drug like compounds, which are an essential part of the research performed in agrochemical and pharmaceutical companies.[6,7]

Organic compounds containing 1,3-amino-oxygenated functional motifs are found 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.[8] 1-Carbamato-alkyl-2-naphthol can be converted to important biologically active 1-aminomethyl-2-naphthol derivatives by carbamate hydrolysis. The hypotensive and bradycardiac effects of these compounds have been established.[9]

## BACKGROUND OF THIS WORK:

For broad range of application, many researchers are involved to develop methodologies for synthesis of 2-amidoalkyl naphthol.

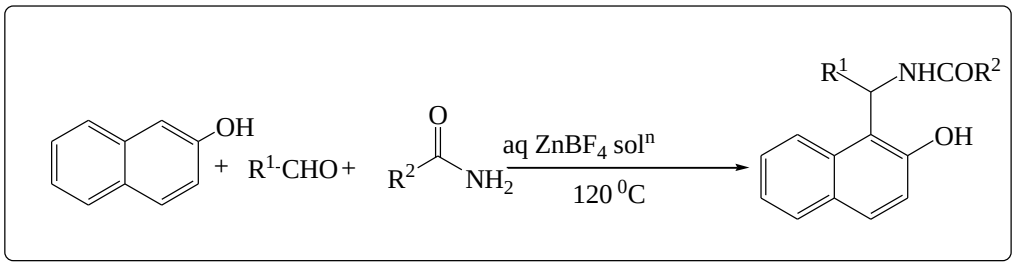
In recent years, the use of economic and green reaction condition has attracted more attention to carry out various organic transformations. Due to a wide range of biological as well as industrial applicability of 2-amidoalkyl naphthol moieties, from earlier days various synthetic routes have been developed to synthesize 2-amidoalkyl naphthol and the derivatives of it. In literature, there are many methods available for synthesis of 2-amidoalkyl naphthol derivatives using different catalyst and reaction conditions e.g. $[Hf(NPf_2)_4]$[10], wet cyanuric chloride (wet TCT)[11], Thiamine hydrochloride[12] , $P_2O_5$[13], Ferric (III) hydrogensulfate[14], silica supported perchloric acid ($HClO_4$-$SiO_2$)[15] , molecular iodine[16] , sulfamic acid[17] etc.

ISSN PRINT 2319 1775 Online 2320 7876

Research paper UGC CARE Listed ( Group -I) Journal Volume 10, Iss 09, 2021

**PRESENT WORK:**

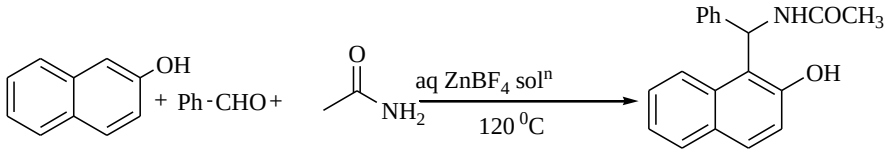
We have developed an efficient, simple and environmentally friendly method for the synthesis of 2-amidoalkyl naphthols by the reaction of 2-naphthol, aldehyde and amide catalyzed by aq. Zinctetrafluoroborate solution (Scheme 1).




**Scheme 1**

Optimization of reaction condition is done by varying catalyst concentration and temperature. The best results were obtain by using 10 mol % of catalyst and at 120$^0$C. The summarization of reaction condition is shown in table 1.

Table 1. Optimization of Reaction Conditions




| Entry | Catalyst | Time (h) | Condition | Solvent | Yields (%)[a] |
|---|---|---|---|---|---|
| 1 | -- | 4 | Heat, 80$^O$C | -- | 05 |
| 2 | -- | 6 | Heat, 130$^O$C | -- | 10 |
| 3 | 10 mol% | 4 | Heat, 120$^o$C | Toluene | 25 |
| 4 | 10 mol% | 4 | Heat, 120$^o$C | DCE | 30 |
| 5 | 10 mol% | 4 | Heat, 120$^o$C | $CH_3CN$ | 52 |
| **6** | **10 mol%** | **4** | **Heat, 120$^O$C** | **--** | **80** |
| 7 | 10 mol% | 4 | Heat, 120$^O$C | DMF | 39 |

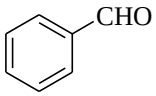
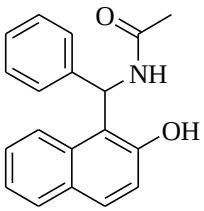
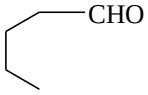
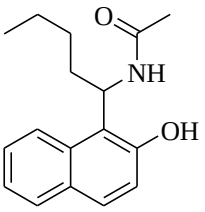
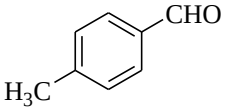
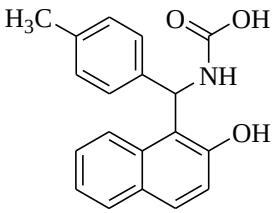
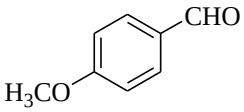
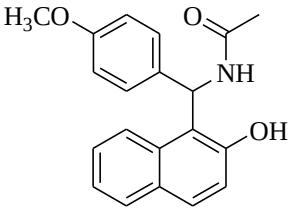
[a] Isolated yields

To explore the scope and limitation of this reaction conditions, we have checked the reaction of 2-naphthol, amide and with a variety of aldehydes. The results are summarized in Table 2.

ISSN PRINT 2319 1775 Online 2320 7876

 UGC CARE Listed ( Group -I) Journal Volume 10, Iss 09, 2021

**Table 2.** Snthesis of amidoalkyl naphthols

| Entry | $R^1$ | $R^2$ | Time (h) | Yield (%)[a] |
|---|---|---|---|---|
| 1 |  |  | 3 | 80 |
| 2 |  |  | 3.5 | 75 |
| 3 |  |  | 3 | 72 |
| 4 |  |  | 4 | 68 |

From the results Table 2 it is quite cleared that this method is uniformly effective for both aromatic and aliphatic aldehydes. Various aromatic aldehydes containing electron-withdrawing and electron-donating substituents show equal effective towards the present protocol.

A probable mechanism for the synthesis of 2-amidoalkyl naphthols is shown in Scheme 2.

## CONCLUSION

We have described a simple, facile, convenient and solvent free method for one pot synthesis of 2-amidoalkyl naphthol derivatives by coupling reaction of various aromatic aldehydes with amide and 2-naphthol using aq. Zinctetrafluoroborate solution as catalst. The eco-friendly experimental condition, ease of reaction, and high yields are significant advantages of the protocol presented here.

**Experimental:** A mixture of amide (1.1 eq), 2-naphthol (1. eq) and aldehyde (1.2 eq) was heated at the appropriate temperature ($120^{O}$ C), in a sealed tube. After completion of reaction (TLC) 30 ml ethyl acetate is added to the reaction mixture, then it was neutralized by washing

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 UGC CARE Listed ( Group -I) Journal Volume 10, Iss 09, 2021

with aqueous solution of sodium hydrogen carbonate (2 x 10ml) and finally with brine solution (2 x 5ml). The organic layer was dried over sodium sulphate and concentrated under reduced pressure. The crude product was purified by re-crystallization from ethanol to furnish respective analytically pure product.

**Acknowledgements:** I am thankful to my student Aakash and Monidipa Department of Chemistry, Jhargram Raj Collegefor their immense support to carry out this work.

## REFERENCES

1. For a monograph on MCRs, *Multicomponent Reactions,* ed. J. Zhu and H. Bienayme, Wiley-VCH, **2005**.
2. For a symposium in print on MCRs, *Tetrahedron Symposia-in-Print,* ed. I. Marek **2005**, 67, 11299.
3. M. D. Burke, S. L. Schreiber, *Angew. Chem. Int. Ed.* **2004**, 43, 46.
4. R. J. Spandl, A. Bender, D. R. Spring, *Org. Biomol. Chem.* **2008**, 6, 1149.
5. L. Weber, *Curr. Med. Chem.* **2002**, 9, 2085.
6. C. Hulme, V. Gore, *Curr. Med. Chem***. 2003**, 10, 51.
7. D. Seebach, J. L. Matthews, *J. Chem. Soc. Chem. Commun.* **1997**, 2015
8. (a) T. Dingermann, D. Steinhilber, G. Folkers, In *Molecular Biology in Medicinal Chemistry*, Wiley-VCH, **2004**; (b) A. Y. Shen, C. L. Chen, C. I. Lin, J. Chin, *Physiol.* **1992**, *35*, 45.
9. M. Hong, C. Cai, W. B. Yi, *Chinese chem. Lett.* **2011**, *22*, 322.
10. G. H. Mahdavinia, M. A. Bigdeli, *Chinese chem. Lett.* **2009**, *20*, 383-386.
11. M. Lei, L. Ma, L. Hu, *Tetrahedron lett***. 2009**, *50*, 6393.
12. G. C. Nandi, S. Samai, R. Kumar, M. S. Singh, *Tetrahedron Lett.* **2009**, *50*, 7220.
13. H. R. Shaterian, H. Yarahmadi, M. Ghashang, *Bioorg. Med. Chem. Lett.* **2008**, *18*, 788.
14. H. R. Shaterian, H. Yarahmadi, M. Ghashang, *Tetrahedron* **2008**, *64*, 1263.
15. B. Das, K. Laxminarayana, B. Ravikanth, B. R. Rao, *J. Mol. Catal. A: Chem.* **2007**, *261*, 180.
16. S. B. Patil, P. R. Sing, M. P. Surpur, S. D. Smant, *Ultrasonics Sonochemistry* **2007**, *14*, 515.