ScienSage

Journal of Advanced Scientific Research

Available online through http://www.sciensage.info

ISSN
0976-9595
Short Communication

SONOCHEMISTRY: SULFAMIC ACID CATALYZED GREEN SYNTHESIS AND CHARACTERIZATION OF SOME β-AMINO CARBONYL LIGANDS CONTAINING CHLORO SUBSTITUENT

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ABSTRACT

In this article, a multicomponent reaction of aromatic ketone, aromatic aldehyde, and aromatic amine that results in the formation of β-amino carbonyl ligands using a Sonochemical method is described. Sulfamic acid, a green and environmental friendly catalyst, catalyzed the reaction. There was no side products produced during the reactions. The present study highlights a fast reaction time, the use of a catalyst, a non-toxic process, simple procedure, and quick product separation. On the basis of ^{1}H NMR spectral analysis, the structures of the synthesized compounds were verified.

Keywords: β-amino carbonyl ligand, Aromatic ketone, Aromatic aldehyde, Mannich reaction.

1. INTRODUCTION

Multicomponent reactions (MCRs) are reactions in which three or more components are interacted to produce preferably one product that includes all of the initial reactants' essential components. By reducing the number of synthetic steps, energy use, and waste generation, MCRs help to meet the requirements of an environmentally friendly procedure [1-10]. As a result, discovering new MCRs and building on those that already exist, is of great interest. The Mannich reaction, for example, is used to produce β-amino carbonyl compounds [11]. The development of carbon-carbon bonds is essential for the formation of organic compounds, and there has recently been a lot of research in this field [12-16]. The Mannich reaction is important in the formation of a broad range of organic molecules [17-24]. The Mannich reaction, which yields industrially and biologically essential β-amino carbonyl compounds, is a classic method for preparing β-amino ketones and aldehydes.

The classical reactions encounter a variety of significant shortcomings, including harsh reaction conditions, extreme side reactions, substrate limits, costly reagents or catalysts, as well as a long reaction performance and low yield. Researchers are working to build eco-friendly ways to address the disadvantages of many conventional methods [25-32]. Sulfamic acid has recently emerged as a potential alternative for a variety of acidic catalysts [33-37]. In the present paper, a facile and efficient method for preparation of β-amino carbonyl compounds by Mannich reaction of acetophenone with aldehyde and aromatic amines in presence of sulfamic acid by Sonochemical approach has been presented.

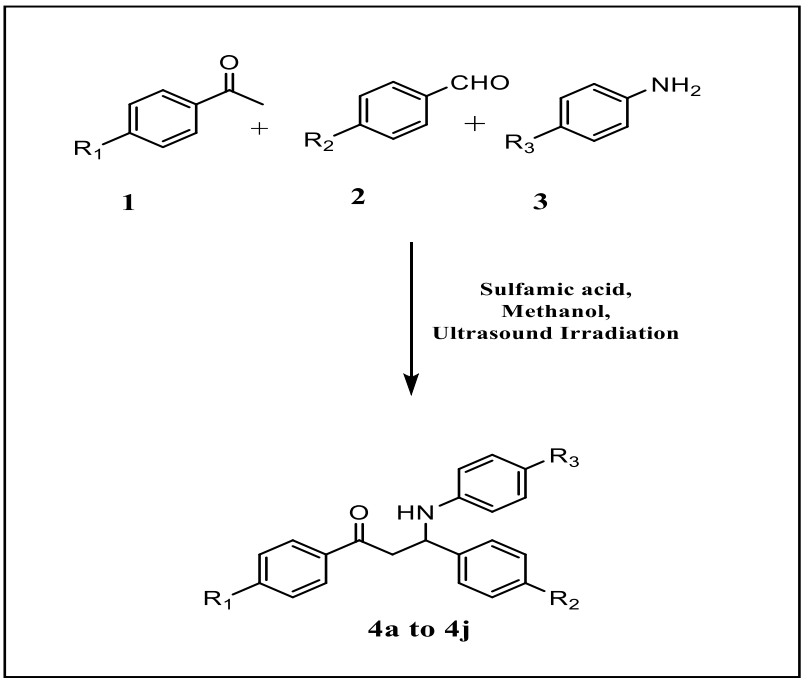
2. MATERIAL AND METHODS

2.1. General Remarks

The AR grade chemicals were purchased and used as received. The reactions were monitored by using thin-layer chromatography on Merck Aluminium TLC plate, silica gel coated with fluorescent indicator F254. Prior to use, all of the glass apparatus were washed and dried in the oven. Solvents were distilled before use.

2.2. Synthesis procedure

To an equimolar mixture (0.01mol) of acetophenones (1), aromatic aldehydes (2), and aromatic amines (3) in conical flask, catalytic amount of sulfamic acid was added. To this mixture, 15 mL methanol solvent was added. The mixture was subjected to ultrasound irradiation until the completion of product. The reaction was monitored by TLC. The crude products were recrystallized from ethyl acetate. The structures of the synthesized compound were affirmed on the basis of 1H NMR spectral analysis.



Scheme 1: Sulfamic acid catalyzed synthesis of β-amino carbonyl ligands

3. RESULTS AND DISCUSSION

The desired compounds were synthesized using a green method involving ultrasound irradiation and a sulfamic acid catalyst. When acetophenone, benzaldehyde, and aniline were exposed to ultrasound irradiation in the presence of a sulfamic acid catalyst, the product was obtained in a surprising amount of time, as indicated by TLC.

3.1. Spectral analysis of selective compounds

3.1.1. 1,3-diphenyl-3-(phenylamino)propan-1-one (4a)

^{1}H NMR (500 MHz, $CDCl_3$): δ 3.51 (m, 2H), 5.01 (t, 1H), 6.62 (d, 2H), 6.69 (m, 1H), 7.12 (m, 2H), 7.23 (m, 1H), 7.32 (m, 2H), 7.47 (m, 4H), 7.55 (m, 1H), 7.99 (m, 2H).

3.1.2. 3-((4-chlorophenyl)amino)-1,3-diphenylpropan-1-one (4b)

^{1}H NMR (500 MHz, $CDCl_3$) 3.47 (d, 1H), 3.47 (d, 1H), 4.90 (m, 1H), 6.67 (d, 2H), 6.62- 6.68(m, 1H), 7.04-7.31 (m, 2H), 7.20 (d, 1H), 7.28 (m, 2H), 6.35 (d, 2H), 7.05(d, 2H), 7.89 (d, 2H).

3.1.3. 1-(4-bromophenyl)-3-phenyl-3-(phenylmino)propan-1-one (4c)

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.48 (m, 2H), 5.04 (t, 1H), 6.63 (t, 2H), 6.70 (t, 1H), 7.12 (t, 2H), 7.23 (d, 1H), 7.31 (t, 2H), 7.48 (d, 2H), 7.57 (d, 2H), 7.80-7.72 (m, 2H).

3.1.4. 3-(4-chlorophenyl)-1-phenyl-3-(phenylmino)propan-1-one(4e)

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.49 (m, 2H), 5.07 (t, 1H), 6.55 (m, 2H), 6.70(m, 1H), 7.16-7.08 (t, 2H), 7.35-7.24 (t, 2H), 7.51-7.38 (m, 4H), 7.68 (t, 1H), 7.93 (d, 2H).

3.1.5. 3-(4-bromophenyl)-1-phenyl-3-(phenylmino)propan-1-one (4i)

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.48 (m, 2H), 5.08 (m, 1H), 6.55 (d, 2H), 6.70 (m, 1H), 7.15-7.07 (m, 2H), 7.45 (d, 2H), 7.48 (m, 4H), 7.64-7.54 (t, 1H), 7.96-7.86 (m, 2H).

3.1.6. 3-((4-bromophenyl)amino)-1,3-diphenylpropan-1-one (4j)

^{1}H NMR (500 MHz, $CDCl_3$) δ 3.49 (m, 2H). 4.95 (t, 1H), 6.46 (d, 2H), 7.15 (d, 2H), 7.24 (m, 1H), 7.35 (t,2H), 7.45 (m,4H), 7.60 (m,1H), 7.97-7.86 (m, 2H). Other reactions were carried out as a result of this exciting result, and all of them produced excellent yields with no side products. The key results of the current synthetic technique were that the study required a short time, simple procedure, excellent yields, and ease of product isolation. Table 1 portrays the

substituent nature, names, time and yield of the of synthesized β-amino carbonyl ligands. Proton magnetic resonance spectral analysis was used to validate the structures of the β-amino carbonyl ligands. All the spectra were clear and showed signals consistent with the product structure, indicating that only one product was formed. Since this technique is so reliable, it could also be used to render other β-amino carbonyl ligands.

Table 1: Name of the of synthesized β-amino carbonyl ligands

Entry	R_1	R_2	R_3	Structure of the product	Time	Yield
4a	H	H	H	1,3-diphenyl-3-(phenylamino)propan-1-one	2	90
4b	H	H	Cl	3-{(4-chlorophenyl)amino}-1,3-diphenylpropan-1-one	2.5	95
4c	Br	H	H	1-(4-bromophenyl)-3-phenyl-3-(phenylamino)propan-1-one	2	96
4d	Cl	H	H	1-(4-chlorophenyl)-3-phenyl-3-(phenylamino)propan-1-one	3	98
4e	H	Cl	H	3-(4-chlorophenyl)-1-phenyl-3-(phenylamino)propan-1-one	2	89
4f	F	H	H	1-(4-fluorophenyl)-3-phenyl-3-(phenylamino)propan-1-one	3	85
4h	H	F	H	3-(4-fluorophenyl)-1-phenyl-3-(phenylamino)propan-1-one	2	87
4i	H	Br	H	3-(4-bromophenyl)-1-phenyl-3-(phenylamino)propan-1-one	1.5	94
4j	H	H	Br	3-{(4-bromophenyl)amino}-1,3-diphenylpropan-1-one	3	95

4. CONCLUSION

In summary, for the synthesis of β-amino carbonyl ligands, an environment friendly multicomponent reaction between aromatic ketones, aromatic aldehydes, and aromatic amines has been employed. Under the Sonochemical approach, sulfamic acid was used as a green catalyst. During the reactions, no side products were produced, which is important. The current method's strengths include a straightforward process and work-up, a low cost, and a benevolent approach, among others.

Funding

No funding was received to carry out research presented in this paper.

Conflict of Interest

Author declares no conflict of interest.

5. ACKNOWLEDGEMENT

The author would like to acknowledge Department of Zoology and Department of Chemistry for providing necessary research facilities to carry out the present research.

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