ISSN No:-2456-2165

Synthesis of Novel α-Glutaryl-N-aryl nitrone (N-(5-Oxopentylidene) Aniline Oxide) and their Antimicrobial Activities

M. Muthu Selvi [1]
PG and Research Department of Chemistry, Government Arts College for Women, Nilakkottai, Affiliated to Mother Teresa Women's University, Kodaikanal, India.624 208 Dindigul, Tamil Nadu, India

M.R. Bhamuni[2], P. Sivadharani[3], S.R. Jayapradha[4]*
PG and Research Department of Chemistry, Government Arts College for Women, Nilakkottai, Affiliated to Mother Teresa Women's University, Kodaikanal, India.624 208 Dindigul, Tamil Nadu, India

Abstract:- Phenyl hydroxyl amine & glutaraldehyde are synthesizing and it has been yielded simple stable form of nitrone. It has been playing an essential part to synthesis the heterocyclic compounds that undergoes the elegant characteristic transformation. Nitrones can be used as probable oxidizing reagents through the renovation of several alkyl halides to ketones and aldehydes with high crop. The novel nitrone is 1,3-Dipole and its used in 1,3-dipolar cycloaddition. The 1,3-dipolar cycloaddition of nitrones with numerous dipolarophiles creates some heterocyclic rings having different sizes and incalculable N-heterocyclic motifs. That has been contains the extensively existed molecules in the biological or pharmaceutical interest. In the present research it has been evaluate a competent method for synthesis of α-Glutaryl-N-aryl nitrone and its employed for their antimicrobial activities and it is confirmed in ^{13}C and ^{1}H NMR techniques.

Keywords:- *α-Glutaryl-N-Aryl Nitrone; Glutaraldehyde; Phenyl Hydroxyl Amine; 1,3 Cycloadditions; Anti-Microbial Activities*

I. INTRODUCTION

Nitrones were tremendously adaptable artificial intermediates and they are of particular standing the reaction of 1,3-dipolar cycloaddition among numerous bond which is important to creating an extensive diversity of five-membered heterocyclic ring systems[1]. At 1963 Mr. Huisgen has made categorization and the concepts of 1,3-dipoles and their reactions of 1,3-dipolar cycloaddition. The initial intramolecular azomethine cyclo addition was established through Regio & stereo controlled technique to synthesis various hetero cyclic compounds in 1976 & reported which is an efficient method and yielding a high crop.[2-4]Nitrones are effective 1,3-dipoles and they could endure voluntarily in cycloaddition process in the midst of electron-deficient olefins to bring into being with substituted isoxazolidines & isoxazolines. There are vital synthetic & environment friendlier methods were created for generating the Five-membered heterocyclic systems & ring through the 1,3-Dipolar cycloaddition reactions. Particularly, versatile Five membered heterocycles ring construction were containing at nitrone. The imperative reasons behind the synthetic application success of nitrones, that it has contained stable & fascinating biological molecules & it can be adaptable intermediates for the synthesis of normal products.[5-7] The present study states that synthesis & antibacterial activity of the derivatives which is derived from nitrone with high crop and remarkable changes in microbial activity.[8] Over the earlier years, 1,3- dipolar cycloaddition of nitrones & Synthesis of isoxazolidines among alkenes have acknowledged significant consideration. Isoxazolidines is a class of influential heterocycles, which is widely used as facade entrant at 1,3-amino-alcohols or a numerous collection of ordinary derivatives and compounds, mainly amino sugar & acids then alkaloids .[9,10]In the recent days there are two methods were widely used to synthesize nitrones (a)condensation reaction of hydroxyl amine with aldehyde and (b) N, *N*-disubstituted amines or secondary amines oxidation. Nitrones were holding the stimulating biological activities such as antioxidant, antifungal, antiviral, and antibacterial.[11,12]

II. EXPERIMENTAL

A. Material and Methods

All chemicals substances were of reagent grade quality and can be used devoid of supplementary distillation (Zn powder, Glutaraldehyde Nitrobenzene and Ammonium chloride). The nitrones were synthesized through the condensation method. Its been remains at longer time periods. Silica gel plates has used to observing the Nitrone reactions in the course of TLC. We have been used Bruker NMR spectra to record the ^{1}H NMR spectra results using $CDCl_3$ as a solvent at 400 MHZ & TMS (diluted Tetramethylsilane) as a internal standard. The same instrument has been used to record ^{13}C NMR spectra on 100 MHz. The coupling constant (J) is shown in Hz.

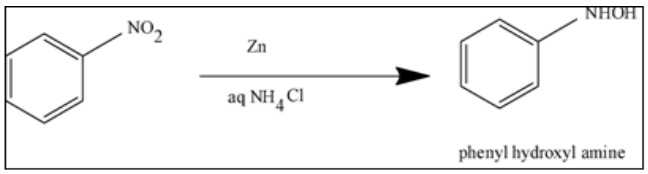
Examined the All synthesized nitrones an under antimicrobial activities against bacterial strains, 20ml of Dimethyl sulfoxide solution has used to dissolved the substance. The agar was used in culture medium and it has been took around 24 hrs. for the growth of strain. The bacteria were spreader over uniformly in across the agar glass plates, all the nitrones were active against specific microorganisms such as Bacillus marisflavi, Pseudomonas aeruginosa, and Exignobacterium indicus. So, our study

ISSN No:-2456-2165

shows all the synthesized nitrones has antimicrobial activities.

III. SYNTHESIS OF PHENYLHYDROXYLAMINE

A conical flask has installed along with mechanical stirrer and thermometer, then add it base chemicals of Ammonium chloride (10g) in 320 ml of H_2O and 16.6 ml of Nitrobenzene in the container and mixes it well. Addition to the mixture put in 23.6 g of Zn powder which is contains 90% purity concerning 15 minutes & the amounts of addition could leading to swiftly increasing the temperature up to 60°-65° C. Then continue the stirring process for further more 15 minutes until that temperature starts reduction. To remove the zinc oxide from the warm reaction mixture, Filter the pump and wash it with 100 ml of hot water.



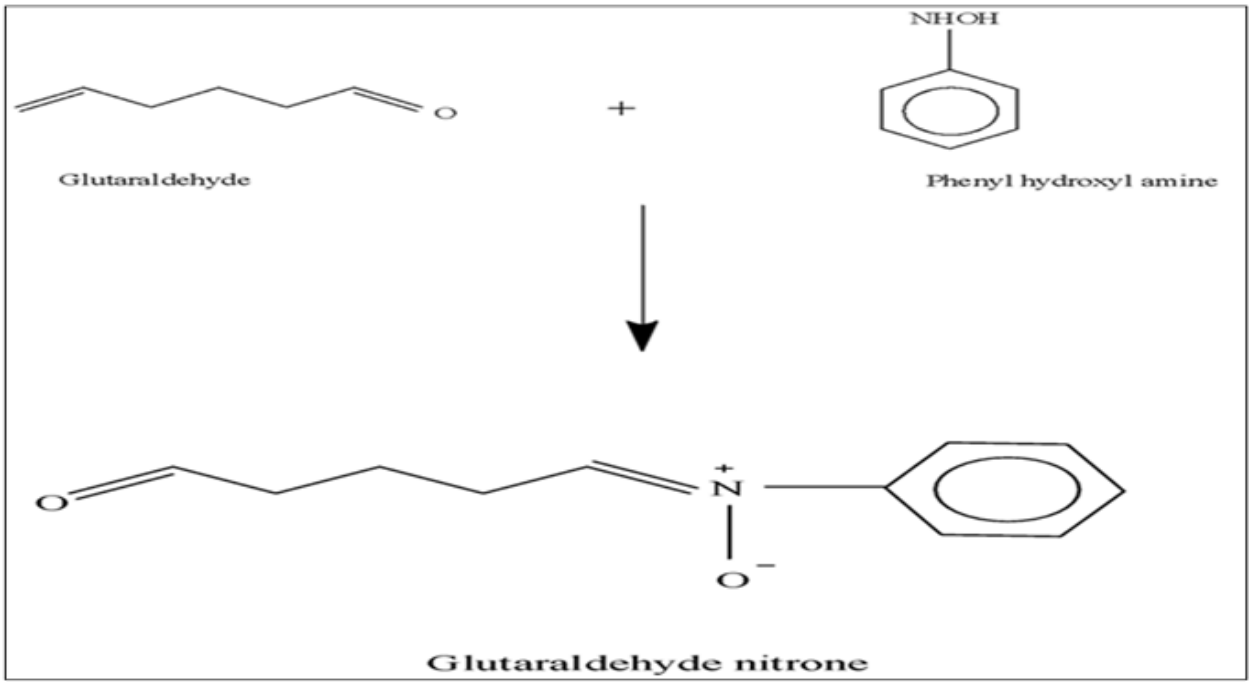
Scheme 1 Synthesis of Phenyl Hydroxylamine

To make certain the max crystallization of the preferred product, placed the remains in the conical flask wet through the mixture with common salt (60g) and cool it with ice cold bath at minimum of one hour. Then strain the pale-yellow crystals of phenyl hydroxyl amine were filtered with suction and drain well. Finally, the phenyl hydroxyl amine was synthesized as shown in scheme 1

IV. RESULTS AND DISCUSSION

A. Synthesis of α-Glutaryl-N-aryl nitrone

The mixture of glutaraldehyde (0.1 m, 10.0 g) was added to phenylhydroxylamine (0.1mol, 10.9g) in the availability of alcohol and it refluxed for 1 hour at room temperature. It could help to develop rudimentary glutaraldehyde nitrone. It was solidified through the cooling and crystallized with solvents on deprived of moisturizer. It was filtered and recrystallized by using ethanol. Through the process we have yielded the untainted synthesized nitrones. The pure form of Glutaraldehyde nitrone was obtained.



Scheme 2 Synthesis of α-Glutaryl-N-aryl nitrone

B. Spectroscopic Data of N-(Oxopentylidene) Aniline Oxide

^{1}H NMR (500 MHz, Chloroform) δ 9.65 (s, 1H), 7.48 –7.42 (m, 2H), 7.35 (s, 1H), 7.31 – 7.19 (m, 2H), 5.14 (s, 1H), 2.75 – 2.63 (m, 2H), 2.51 – 2.37 (m, 2H), 1.98 – 1.86 (m, 2H). ^{13}C NMR (125 MHz, Common NMR Solvents) δ 198.49 (s), 158.55 (s), 134.59 (s), 129.31 – 128.92 (m), 122.34 – 121.94 (m), 38.77 (s), 23.13 (s), 22.60 (s).

C. Antimicrobial study of N-(Oxopentylidene) Aniline Oxide

Dimethyl sulfoxide (DMSO) were helping to enhance the bacteria growth. So Dimethyl sulfoxide (DMSO) & N-(Oxopentylidene)aniline oxide were dissolved and stored in the agar plates to monitor the growth. It was added in the combination of 20 ml & 20 mg respectively in the agar plates. Incubation method were used to observe the nitrone growth. The nitrone spreads well in agar plates after the 24 hrs. of incubation. Evidenced the Antibacterial activities in the synthesized nitrone against 3 bacterial strains shown in Table. 1. The values are detected with the explicit organisms like Bacillus marisflavi [13mm], Pseudomonas aeruginosa [14mm], and Exignobacterium indicus [18mm]. Fig. 1 represents the organisms of α-Glutaryl-N-aryl nitrone.

ISSN No:-2456-2165

Table 1 Values and Organisms of α-Glutaryl-N-aryl nitrone

Organisms	α-Glutaryl-N-aryl nitrone
Bacillus marisflavi	**13 mm**
Pseudomonas aeruginosa	**14 mm**
Exignobacterium indicus	**18 mm**

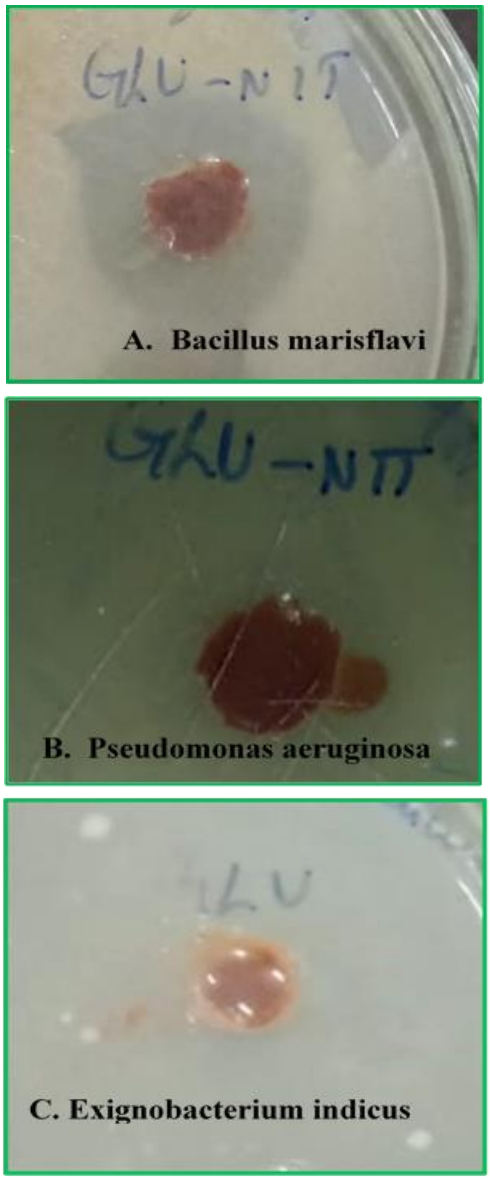


Fig 1 [A], Bacillus Marisflavi [B] Pseudomonas Aeruginosa [C]Exignobacterium Indicus of α-Glutaryl -N-aryl nitrone

V. CONCLUSIONS

With reference to the above study we have been confirming that acknowledged a excellent vintage of novel nitrone compound through the condensation reaction & establishing the general methodology applicable to the synthesis of phenyl hydroxyl amine like highly substituted different aldehydes. Confirmation of substituent's effect of the phenyl hydroxyl amine has also been analyzed. The above proposal has confirming that nitrone construction fashioned healthy. The nitrone structure construction has been confirmed through antimicrobial studies and ^{1}H NMR, ^{13}C NMR spectra. The method of synthesizing nitrones is an environment friendly, cost effective, ease & comfort to follow. The great reactivity and permanency of the nitrones make them outstanding preliminary materials for an assortment of products. By considering the above outlook, it can be decided that the usefulness of nitrones will unquestionably increase in adjacent forthcoming.

REFERENCES

[1]. Graeme M. Robertson, in Comprehensive Organic Functional Group Transformations, 1995
[2]. (a) Huisgen, R. Angew. Chem., Int. Ed. Engl. 1963, 2, 565;
(b) Confalone, P. N.; Huie, E. M. Org. React. 1988, 36, 1; (c) Carruthers, W. Cycloaddition Reactions in Organic Synthesis; Pergamon: Oxford, U.K., 1990; Chapter 6, p 269.
[3]. (a) Padwa, A. In Comprehensive Organic Synthesis; Trost, B. M., Fleming, I., Eds.; Pergamon: Oxford, U.K., 1991; Vol. 4, p 1069;
(b) Padwa, A.; Weingarten, M. D. Chem. Rev. 1996, 96, 223;
(c) Synthetic Applications of 1,3-Dipolar Cycloaddition Chemistry Toward Heterocycles and Natural Products; Padwa, A., Pearson,W. H., Eds.; Wiley: New York, 2003.
[4]. (a) Karlsson, S.; Ho¨gberg, H.-E. Org. Prep. Proc. Int. 2001, 33, 103; (b) Gothelf, K. V.; Jørgensen, K. A. Chem. Rev. 1998, 98, 863; (c) Koumbis, A. E.; Gallos, J. K. Curr. Org. Chem. 2003, 7, 771.
[5]. (a) R. Huisgen, in: A. Padwa (Ed.), 1,3-Dipolar Cycloaddition Chemistry, vols. 1 and 2, Wiley–Interscience, New York, 1984; (b) A. Padwa, in: B.M. Trost, I. Fleming (Eds.), Comprehensive Organic Synthesis, vol. 4, Pergamon, Oxford, 1991, p. 1069
[6]. A. Padwa, M.D. Weingarten, Chem. Rev. 96 (1996) 223–269.
[7]. (a) K.V. Gothelf, K.A. Jorgensen, Chem. Rev. 98 (1998) 863–909; (b) P. Grunanger, P. Vita-Finzi, Isoxazoles, Wiley, New York, 1991.
[8]. Bhaskar Chakraborty Sikkim Government College (NBBDC), Gangtok 737102, Sikkim, India
[9]. Carda, M.; Portoles, R.; Murga, J.; Uriel, S.; Marco, J. A.; Domingo, L. R.; Zaragoza, R. J.; Roeper, H. J. Org. Chem. 2000, 65, 7000–7009.
[10]. (a)Ravi Kumar, K. R.; Mallesha, H.; Rangappa, K. S. Synth. Commun. 2003, 33, 1545–1555.
[11]. Habib Mosbah1 & Hassiba Chahdoura1 & Asma Mannai 2 & Mejdi Snoussi 2 & Kaïss Aouadi3
[12]. Rui M. V. Abreu4 & Ali Bouslama5 & Lotfi Achour 1 & Boulbaba Selmi