

SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL STUDIES OF 7-ETHYL-2, 4-BIS(4-METHOXY-PHENYL)-3-AZA-BICYCLO[3.3.1]NONAN-9-ONE

**S. Mohamed Rabeek¹, M. Seeni Mubarak^{*1}, H.Mohamed Kasim Seit¹, R. Kathirvel¹,
M. Sathyanarayanan¹**

¹PG and Research Department of Chemistry, Jamal Mohamed College (Autonomous)
Affiliated Bharathidasan University, Tiruchirappalli- 620020, Tamilnadu, India.

Article Received on
10 Nov 2014,

Revised on 19 Dec 2014,
Accepted on 10 Jan 2015

***Correspondence for**

Author

M. Seeni Mubarak

PG and Research
Department of
Chemistry, Jamal
Mohamed College
(Autonomous) Affiliated
Bharathidasan
University,
Tiruchirappalli,
Tamilnadu, India
rafeeqchem@yahoo.com

ABSTRACT

The newly synthesized 7-ethyl-2,4-bis(4-methoxy-phenyl)-3-aza-bicyclo[3.3.1]nonan-9-one. The compound has been derived by the condensation of 4-ethylcyclohexanone and 4-methoxybenzaldehyde using ammonium formate. The structure of the synthesized compound was elucidated by spectral studies such as IR, ¹H, ¹³C NMR and elemental analysis. The newly synthesized compound was screened for its antimicrobial activity against staphylococcus aureus, escherichiacoli, pseudomonas aerugionosa, aspergillusnigar and mucor.

Keywords: 4-ethylcyclohexanone, 4-methoxybenzaldehyde, Spectral studies, Antimicrobial activity.

INTRODUCTION

Heterocyclic compounds represent an important class of biologically active molecules. A deep study of literature review showed that the study of reaction like Claisen-Schmidt, Mannich¹ and aldol condensation. Owing to their pharmacological activity of such as antibacterial², antifungal³, anti-inflammatory⁴, antimalaria, cytotoxicity and anticancer⁵ activities. In the present study we have reported for its antibacterial, antifungal activities. It has been planned in this work to synthesis a novel type of compounds using cyclic ketones, aldehydes, and amines.

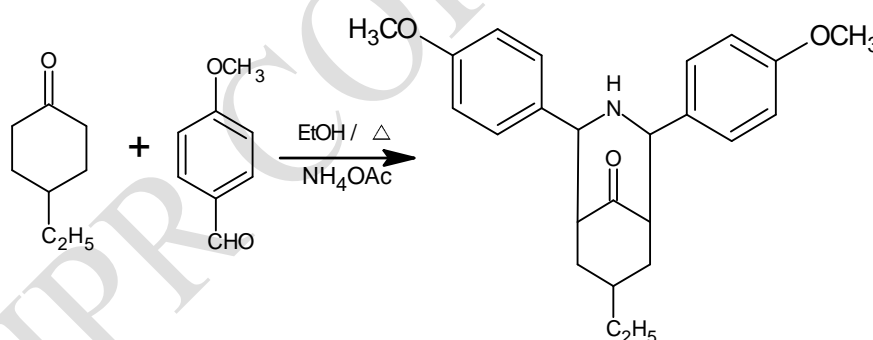
EXPERIMENTAL

Melting points were determined with open capillary and are uncorrected. Proton NMR spectra were taken in DMSO and recorded at 400 MHz in Burker and ^{13}C NMR spectra were taken in DMSO and recorded in 300 MHz in Burker. Chemical shifts were measured in ppm with respect to TMS. FTIR spectra recorded on instrument Simadzu 2100 S and Perkin Elmer BX.

MATERIALS AND METHODS

4-ethylcyclohexanone (0.01mol), ammonium acetate (0.01mol), 4-methoxybenzaldehyde (0.02mol) in a RB flask containing ethanol (10ml). The mixture is refluxed at 60-70 °C in a water bath with occasional shaking until the colour changes into red orange. The solution is cooled, and then ether (50ml) is added. The filtered solution is transfer into conical flask. The yellow colour precipitate formed is filtered and dried. Then the product is recrystallised with ethanol and crystal form of product obtained is dried. The melting point is 160°C.

(4-ethylcyclohexanone and ammonia acetate supplied by E.merck were used as such. 4-methoxybenzaldehyde were supplied by BDH. Ethanol was distilled twice to get maximum alcohol content. Silicagel.G supplied by BDH was used to prepare TLC plates)



Scheme I

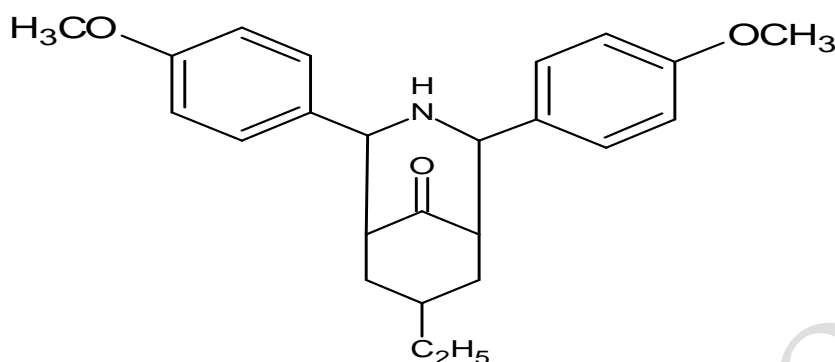
RESULT AND DISCUSSION

SPECTRAL DATA

IR (KBr): 3059 cm^{-1} (NH), 2997 cm^{-1} (aromatic C-H), 2924 cm^{-1} (aliphatic C-H), 1664 cm^{-1} (C=O), 1145 cm^{-1} (C-O-C); ^1H NMR(400 MHz, DMSO): δ 7.53-7.50 and 7.04-7.01 (8H, aromatic proton), δ 3.80 (3H OCH_3 proton), δ 2.50 (1H NH proton); ^{13}C NMR (DMSO): Chemical shift value 159 ppm(C=O), 135 ppm(Ipso carbon), 133 ppm(carbons of phenyl ring), 114 ppm(substituted O- CH_3 carbon), 55 ppm(O- CH_3), 34 ppm(C_7).

Based on the above spectral data the compound is identified as

7-ethyl-2,4-bis(4-methoxy-phenyl)-3-aza-bicyclo[3.3.1]nonan-9-one and the given structure



ANTIMICROBIAL ACTIVITY

The compound was dissolved in DMSO. The filter paper disks were soaked with different solution of the compound dried and then placed in petri dish previously seeded with test organism *E-coli*, *S-aureus*, *P. aeruginosa*, *A-nigar* and *mucor*. The plates were incubated for 24h at 37 °C and incubation zone around each disc was measured the biological activities of the synthesized compound was studied against the *E-coli*, *S-aureus*, *P. aeruginosa*, *A-nigar* and *mucor* using agar plates technique at concentration of 1mg/ml in DMSO. The compound show inhibition zone diameter ranges between 10-15 mm as shown in table. The compound show greater antimicrobial activity towards *E-coli* and antifungal activity towards *A.nigar* while all other showed moderate activity against all the bacterial and fungal cultures.

Table No.1: Results of Antimicrobial Activity

Antimicrobial			Antifungal	
E-coli	S-aureus	P.aeruginosa	A.nigar	Mucor
38	35	35	35	32

(Reference standard Ciprofloxin 5µ/g disk for bacteria; Nystain 100 µ/g disk for fungi)

CONCLUSION

In this part the synthesis of new cyclic Mannich base is explained very clearly with the reaction the structure of the synthesized compound is analyzed by spectral data and antimicrobial studies.

ACKNOWLEDGEMENT

Authors are thankful to the Principal and Management, Jamal Mohamed College (Autonomous), Tiruchirappalli, Tamilnadu for their supporting and encouragement.

REFERENCES

1. Pandeya SN., Sriram D., Nath G., Declerq E. *Eur.J.Pharm sci.* 1999; 9:25-31
2. Mobio IG., Soldatenkov AT., Federov VO., Ageev EA., Sergeeva ND., Lin S., Stashenku EE., Prostakov NS., Andreeva EL. *Khim. Farm. Z.* 1989; 23: 421.
3. Katritzky AR., Fan WJ. *J. Org.Chem.* 1990; 55 (10): 3205-3209.
4. Dimmock JR., Padmanilayam MP., Puthucode RN., Nazarali AJ., Motaganahalli NL., Zello GA., Quail JW., Oloo EO., Kraatz HB., Prisciak JS., Allen TM., Santos CL., Balzarini J., De Clercq E., Manavathu EK. *J. Med. Chem.* 2001; 44 (4): 586-593.
5. El-Subbagh HI., Abu-Zaid SM., Mahran MA., Badria FA., Al-obaid AM. *J. Med. Chem.* 2000; 43:2915.
6. Seeni Mubarak M., et al., *Oriental J. Chem.*, 2011; 27(1): 333.
7. Fazal Mohamed M.I., Seeni Mubarak M. *Indian. J. Heterocyclic Chem.* 2008; 18:19.
8. Afshan Banu, Vasundhara D.E., Ravi S., Lamani, *J.Saudi Chem Soc.*, 2013; 17:211-217.
9. Sireesha B., Krishna Rao B., Sarala Devi Ch., Raghavaiah P., *Indian J. Chem.* 2008; 47B: 592
10. Baliah V., Ekambaram A., Govindarajan TS. Condensation of acetone with aldehydes and ammonia. *Letters to the Editor. Curr. Sci.* 1954; 23: 264.