iJRASET
International Journal For Research in Applied Science and Engineering Technology

# INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

**Volume:** 5 **Issue:** XI **Month of publication:** November 2017

**DOI:**

www.ijraset.com

Call: 08813907089 | E-mail ID: ijraset@gmail.com

International Journal for Research in Applied Science & Engineering Technology (IJRASET)
*ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor:6.887*
*Volume 5 Issue XI November 2017- Available at www.ijraset.com*

# Synthesis of Schiff's Base Derivatives Using Water as Solvent.(A Green Methodology)

Ajmal R. Bhat[1], M. Hussain Wagay[2]
[1,2]*Department of Chemistry, Sant Baba Bhag Singh University, Jalandhar, Punjab 144030*

***Abstract:*** ***The synthetic work has been done by using the various substituted aromatic aldehydes and ethylene diamine under mild conditions using water as green solvent. On completion of reaction the product was separated. dried, and recrystallized from ethanol to obtained 75-90 % yield. All these advantages make this methodology an alternative platform to the conventional method and also make it significant under the umbrella of environmentally greener and safer processes.***
***Keywords: Aromatic Aldehydes, ethylene diamine, Water as green solvent.***

## I. INTRODUCTION

Green chemistry is defined as invention, design, development and application of chemical products and processes to reduce the use and production of substances which may cause harm to health and environment. Green chemistry is the branch of chemistry that involves tools, techniques and technologies. It is helpful to chemists and chemical engineers in research, development and production, for development of more eco-friendly and efficient products which may also have significant financial benefits. It is going to now become an essential tool in the field of synthetic chemistry [1]. It is a new way of looking at organic synthesis and the design of drug molecules, offering important environmental and economical advantages over traditional synthetic processes [2]. The recent interest in green chemistry has posed a new challenge for organic synthesis in that new reaction conditions need to be found which reduce the emission of volatile organic solvents and the use of hazardous toxic chemicals [3]. They improve selectivity, reduce reaction time, and simplify separation and purification of products than the conventional methods [4].

The chemistry of carbon-nitrogen double bond has played a vital role in the progress of chemical sciences. Due to presence of a lone pair of electrons on the nitrogen atom and general electron donating character of the double bond, these compounds have found very large applications in the field of chemistry.Schiff's bases are a class of compounds containing the Azomethine functional group [5] which is a carbon-nitrogen double bond (>C=N-), nitrogen being attached to an alkyl or aryl group, but not hydrogen and represented by the general formula $R_3R_2C=NR_1$. These compounds are named after Hugo Schiff and are represented by the general structure as below:

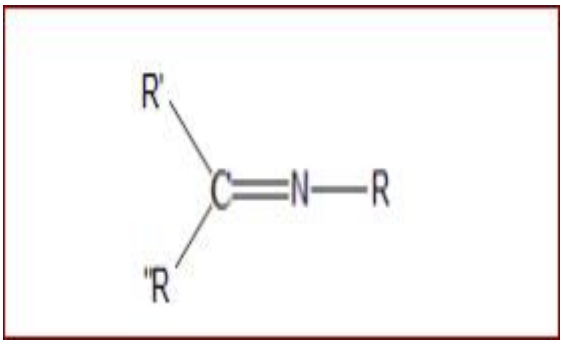
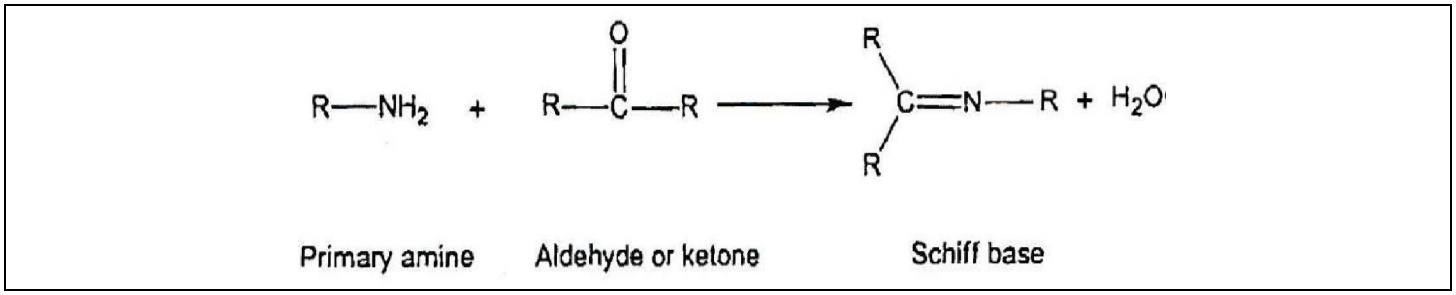



Figure 1.1: General structure of Schiff's base

Where 'R' stands for a phenyl or an alkyl group which makes the Schiff bases a stable imine.

They are the condensed products of aldehydes or ketenes and were first reported by Hugo Schiff in 1864 [6]. Originally, the classical synthetic route for synthesis of Schiff bases was reported by Schiff which involves condensation of primary amines with carbonyl compounds [7] under isotropic distillation with the simultaneous removal of water. (Scheme 1.1)

©IJRASET (UGC Approved Journal): All Rights are Reserved



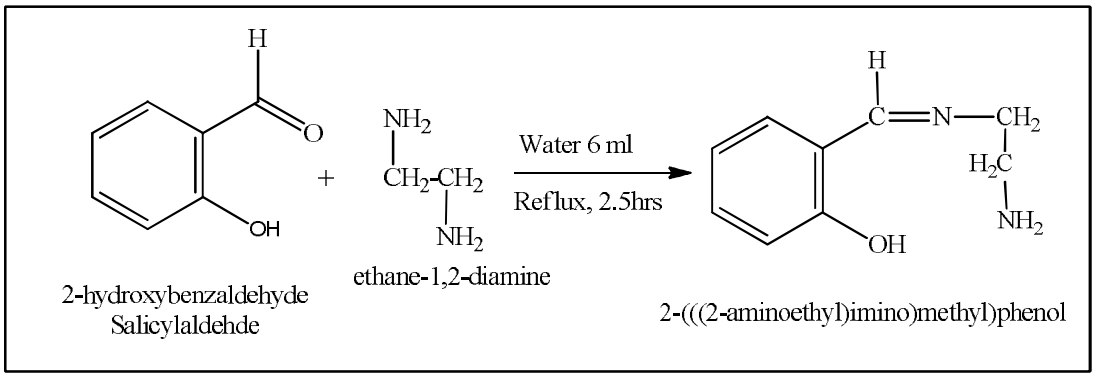

Scheme 1.1: Formation of Schiff's base.

Where R, may be an alkyl or an aryl group. Schiff's bases that contain aryl substituent's are substantially more stable and more readily synthesized, while those which contain alkyl substituent's are relatively unstable. In conclusion, we describe a green and efficient method for the synthesis of Schiff bases in water media. The advantages are easy workup, facile conditions, fast reaction rates, good yields, and selectivity of the reaction make the present methodology attractive.

## II. EXPERIMENTAL

### A. *Synthesis of Salicylaldehyde derivatives*

Take 1.06ml (0.01mm) salicylaldehyde and 0.7ml ethylene diamine in a round bottomed flask. Dissolve the mixture in 6ml of water as a solvent and shake it so that the mixture gets completely dissolved. Reflux the mixture under a condenser for approximately about 2.5hours at a temperature of about $70^0$C till the reaction is completed. On completion the product is separated. Filter off the compound and wash several times with cold water. Recrystallize the compound using ethanol as solvent and then filter the final product. Dry and weigh the final product **(Scheme 2.1).**




Scheme 2.1:General reaction for synthesis of 2-Hydroxy derivative of Schiff base.

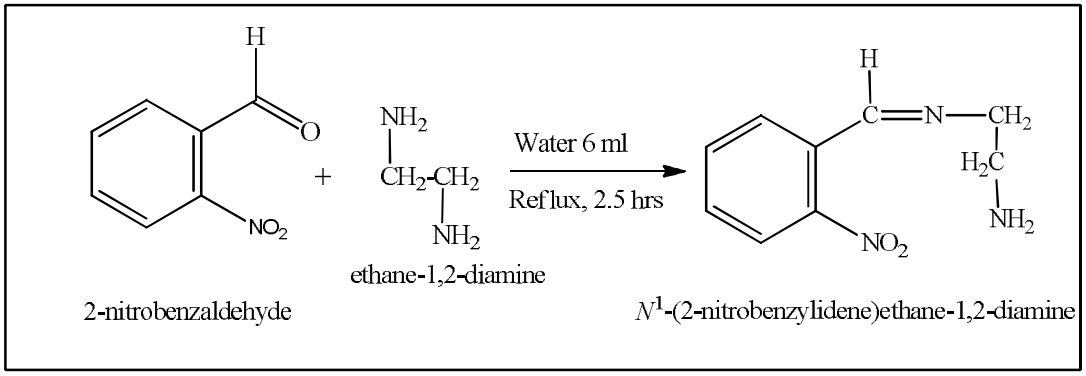
| | |
|---|---|
| Yield | 2.33g |
| Percentage Yield | 69.13% |
| Colour | Yellowish solid powder |
| Solubility | DMSO |
| Melting point | $120^0$C |
| $^1$HNMR analysis | Please see the results and discussion |

### B. *Synthesis of 2-Nitrobenzaldehyde derivatives*

Take 1.51g(0.01mm) of 2-nitrobenzaldehyde and 0.7ml of ethylene diamine in a round bottomed flask. Dissolve the mixture in 6ml of water as a solvent and shake it so that the mixture gets completely dissolved. Reflux the mixture under a condenser for approximately about 2.5hours at a temperature of about $70^0$C till the reaction is completed. On completion of the reaction the

©IJRASET (UGC Approved Journal): All Rights are Reserved

product is separated. Filter off the compound and wash several times with cold water. Recrystallize the compound using ethanol as a solvent and then filter the inal product. Dry and weigh the final product (Scheme 2.2).

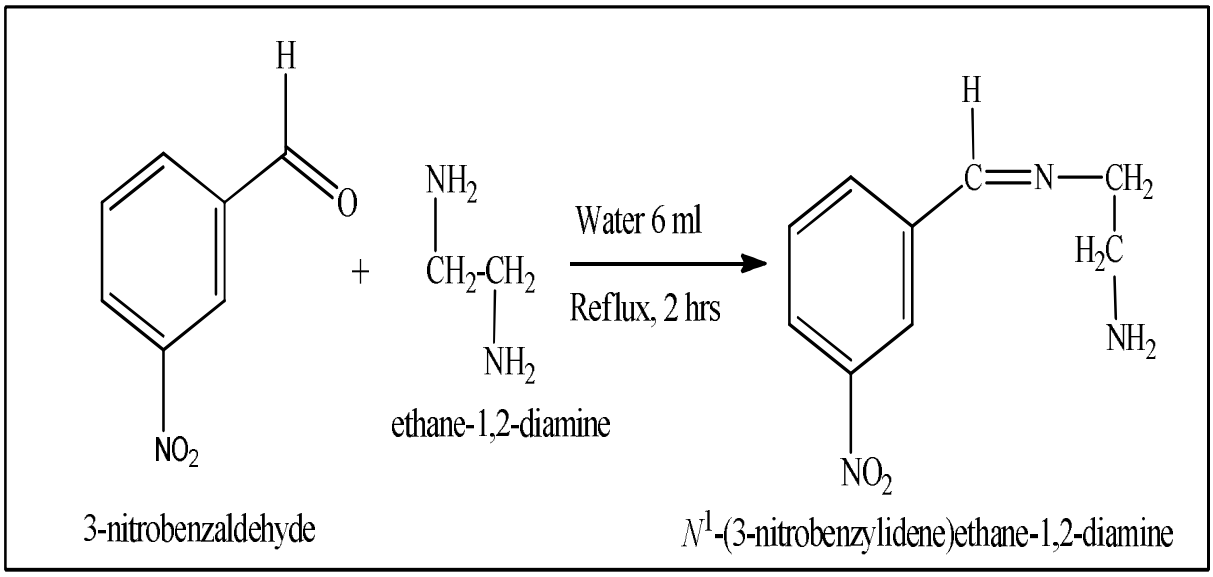



Scheme 2.2: General reaction for synthesis of 2-Nitro derivative of Schiff base.

| | |
|---|---|
| Yield | 2.37g |
| Percentage Yield | 70.32% |
| Colour | Light brown |
| Solubility | DMSO |
| Melting point | $118^0$C |
| $^1$HNMR analysis | Please see the results and discussion |

*C. Synthesis of 3-Nitrobenzaldehyde derivatives.*

Take 1.51g(0.01mm) of 3-nitrobenzaldehyde and 0.7ml of ethylene diamine in a round bottomed flask. Dissolve the mixture in 6ml of water as a solvent and shake it so that the mixture gets completely dissolved. Reflux the mixture under a condenser for approximately about 2hours at a temperature of about $70^0$C. On completion of the reaction the product is separated. Filter offthe compound and wash several times with cold water. Recrystallize the compound using ethanol as a solvent and then filter the final product. Dry and weigh the final product (Scheme 2.3).



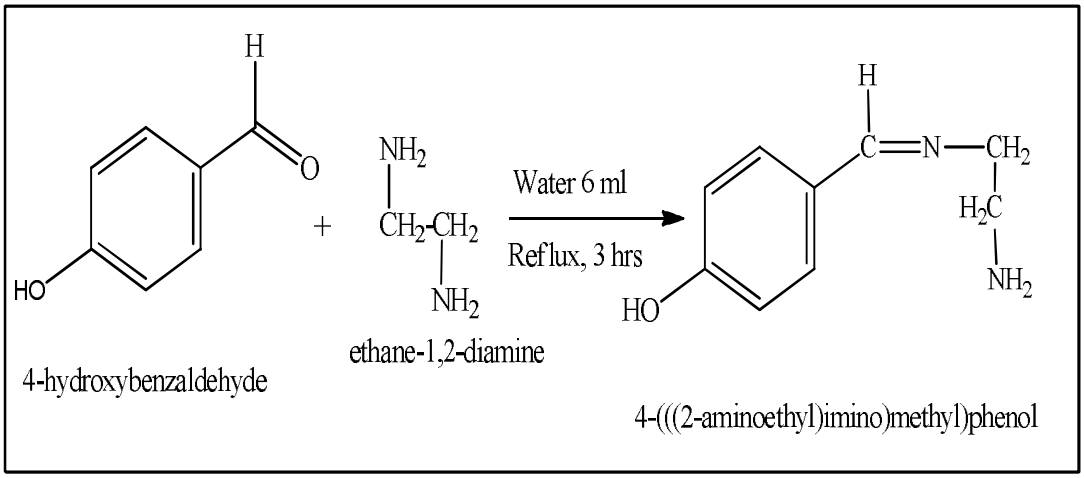

Scheme 2.3: General reaction for synthesis of 3-Nitro derivative of Schiff base.

©IJRASET (UGC Approved Journal): All Rights are Reserved

| | |
|---|---|
| Yield | 2.21g |
| Percentage Yield | 65.57% |
| Colour | White solid |
| Solubility | DMSO |
| Melting point | $161^0$C |
| $^1$HNMR analysis | Please see the results and discussion |

*D. Synthesis of 4-Hydroxybenzaldehyde derivatives.*

Take 1.29g(0.01mm) of 4-hydroxybenzaldehyde and 0.7ml of ethylene diamine in a round bottomed flask. Dissolve the mixture in 6ml of water as a solvent and shake it so that the mixture gets completely dissolved. Reflux the mixture under a condenser for approximately about 3hours at a temperature of about $70^0$C. On completion of the reaction the product is separated. Filter off the compound and wash several times with cold water. Recrystallize the compound using ethanol as a solvent and then filter the final product. Dry and weigh the final product (Scheme 2.4).



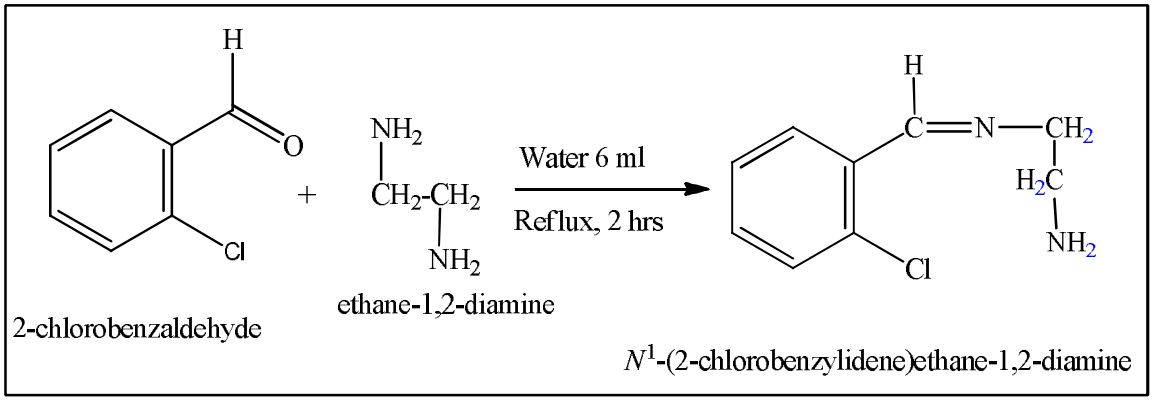

Scheme 2.4: General reaction for synthesis of 4-Hydroxy derivative of Schiff base.

| | |
|---|---|
| Yield | 2.51g |
| Percentage Yield | 74.48% |
| Colour | Light brown |
| Solubility | DMSO |
| Melting point | $190^0$C |
| $^1$HNMR analysis | Please see the results and discussion |

*E.* Synthesis of 2-Chlorobenzaldehyde derivatives.

Take 1.12ml (0.01mm) of 2-chlorobenzaldehyde and 0.7ml of ethylene diamine in a round bottomed flask. Dissolve the mixture in 6ml of water as a solvent and shake it so that the mixture gets completely dissolved. Reflux the mixture under a condenser for about approximately 2hours at a temperature of about $70^0$C. On completion of reaction the product is separated. Filter off the compound and wash several times with cold water. Recrystallize the compound using ethanol as a solvent and then filter the final product. Dry and weigh the final product (Scheme 2.5).

©IJRASET (UGC Approved Journal): All Rights are Reserved




Scheme 2.5: General reaction for synthesis of 2-Chloro derivative of Schiff base.

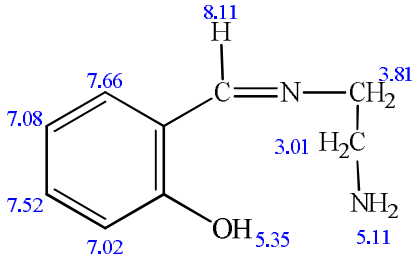
| | |
|---|---|
| Yield | 2.19g |
| Percentage Yield | 65.17% |
| Colour | Yellowish white solid |
| Solubility | DMSO |
| Melting point | $90^0$C |
| $^1$HNMR analysis | Please see the results and discussion |

Table-2: Synthesis of Schiff's base derivatives using water as solvent.

| S.No. | Aryl aldehydes | Time | %yield | Melting point ($^O$C) |
|---|---|---|---|---|
| 1. | Salicylaldehyde | 2.5hrs | 69.13 | 120 |
| 2. | 2-Nitrobenzaldehyde | 2.5hrs | 70.32 | 118 |
| 3. | 3-Nitrobenzaldehyde | 2hrs | 65.57 | 161 |
| 4. | 4-Hydroxybenzaldehyde | 3hrs | 74.48 | 190 |
| 5. | 2-Chlorobenzaldehyde | 2hrs | 65.17 | 90 |

*F. Spectral data of Synthesised compounds.*

*1) Compond 1:*




2-(((2-aminoethyl)imino)methyl)phenol

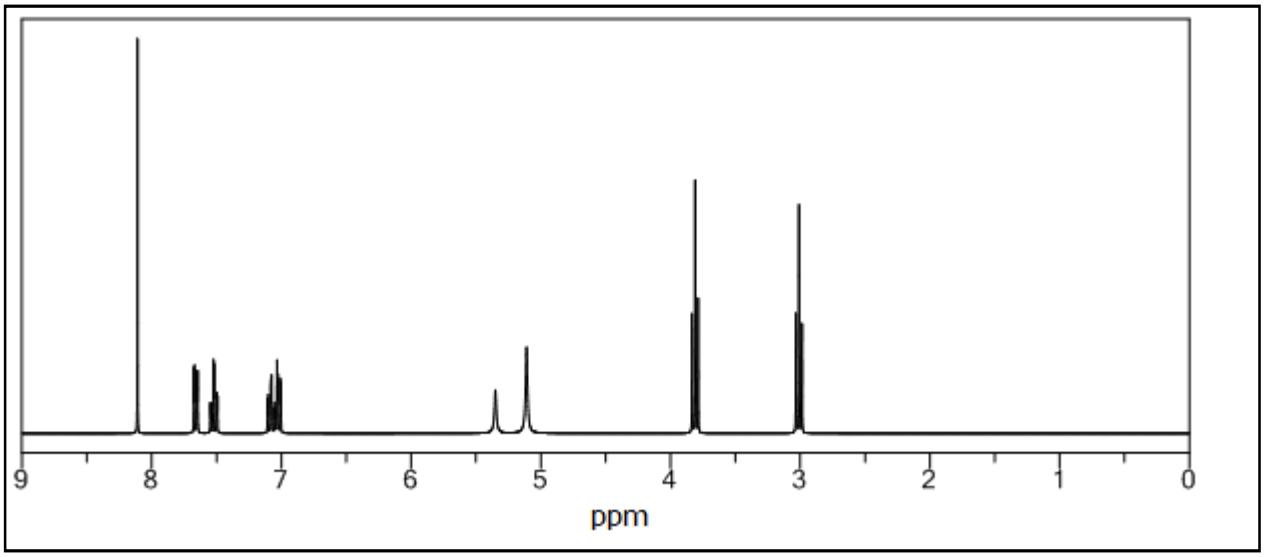



Figure 2.1: [1]HNMR spectra of 2-(((2-aminoethyl)imino)methyl)phenol.

*G. Result And Discussion*

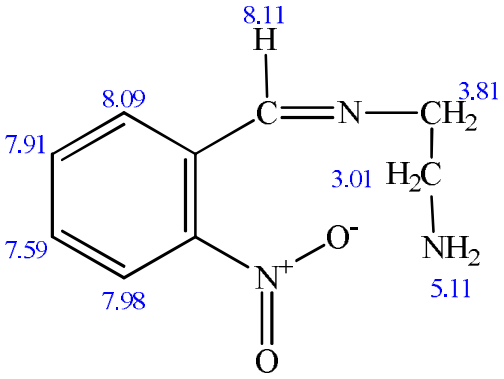
Total number of protons= 12

-$NH_2$= 5.11 ppm
-$CH_2$= 3.81 ppm
-$CH_2$= 3.01 ppm
-CH= 8.11 ppm
-OH= 5.35 ppm
-Aromatic protons= 7.02, 7.52, 7.08, 7.66 ppm

| Node | Shift | Base + Inc. | Comment (ppm rel. to TMS) |
|---|---|---|---|
| OH | 5.35 | 5.00 | aromatic C-OH |
| | | 0.35 | general corrections |
| NH2 | 5.11 | 2.00 | amine |
| | | 3.11 | general corrections |
| CH | 7.02 | 7.29 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | -0.53 | 1 -O from 1-benzene |
| | | 0.26 | general corrections |
| CH | 7.66 | 7.62 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | -0.17 | 1 -O from 1-benzene |
| | | 0.21 | general corrections |
| CH | 7.52 | 7.29 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | -0.17 | 1 -O from 1-benzene |
| | | 0.40 | general corrections |
| CH | 7.08 | 7.29 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | -0.44 | 1 -O from 1-benzene |
| | | 0.23 | general corrections |
| CH2 | 3.81 | 1.37 | methylene |
| | | 2.22 | 1 alpha -N=C-1:C*C*C*C*C*C*1 |
| | | 0.22 | 1 beta -N |
| CH2 | 3.01 | 1.37 | methylene |
| | | 1.32 | 1 alpha -N |
| | | 0.32 | 1 beta -N=C-1:C*C*C*C*C*C*1 |
| H | 8.11 | 8.11 | benzylidenimin |
| | | 0.00 | 1 -C |

©IJRASET (UGC Approved Journal): All Rights are Reserved

*1) Compound 2*




$N^1$-(2-nitrobenzylidene)ethane-1,2-diamine

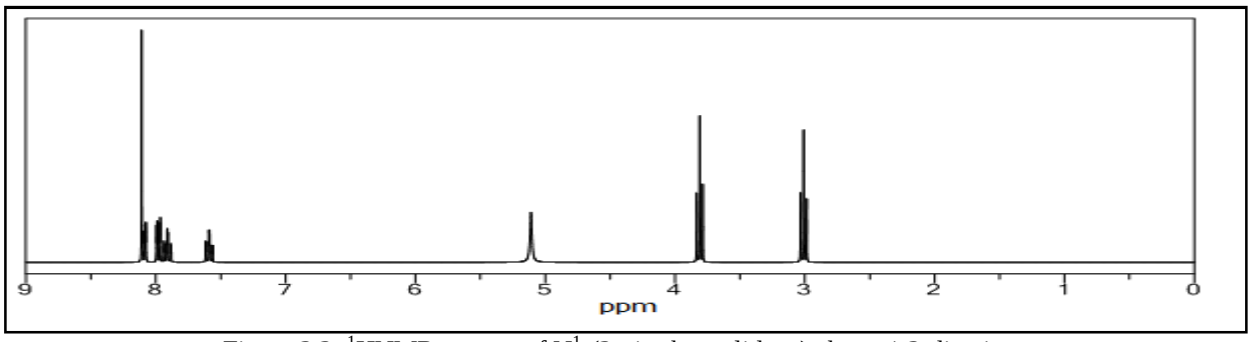



Figure 2.2: $^1$HNMR spectra of $N^1$-(2-nitrobenzylidene)ethane-1,2-diamine.

*H. Result and discussion*

Total number of protons= 11

-$NH_2$= 5.11 ppm

-$CH_2$= 3.01 ppm

-$CH_2$= 3.81 ppm

-CH= 8.11 ppm

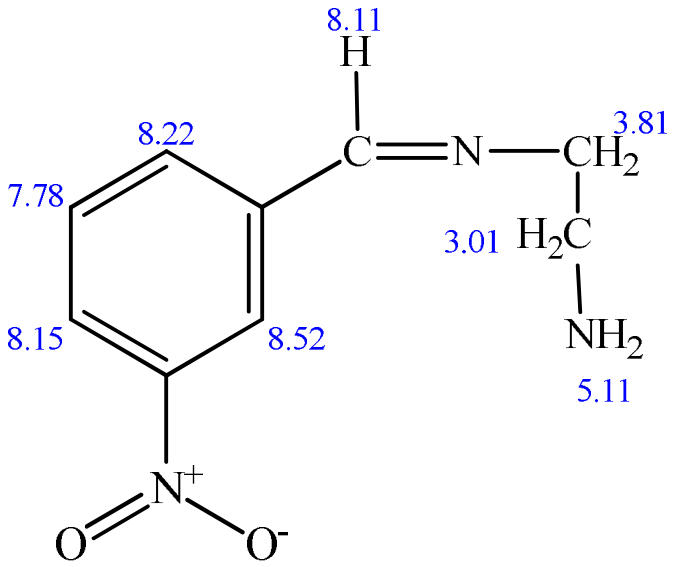
-Aromatic protons= 7.98, 7.59, 7.91, 8.09 ppm.

```
Node      Shift     Base + Inc.    Comment (ppm rel. to TMS)

NH2  5.11              2.00        amine
                       3.11           general corrections
CH   7.98              7.29        benzylidenimin
                       0.00           1 -C
                       0.93           1 -N(=O)=O from 1-benzene
                      -0.24           general corrections
CH   8.09              7.62        benzylidenimin
                       0.00           1 -C
                       0.26           1 -N(=O)=O from 1-benzene
                       0.21           general corrections
CH   7.59              7.29        benzylidenimin
                       0.00           1 -C
                       0.26           1 -N(=O)=O from 1-benzene
                       0.04           general corrections
CH   7.91              7.29        benzylidenimin
                       0.00           1 -C
                       0.39           1 -N(=O)=O from 1-benzene
                       0.23           general corrections
CH2  3.81              1.37        methylene
                       2.22           1 alpha -N=C-1:C*C*C*C*C*C*1
                       0.22           1 beta -N
CH2  3.01              1.37        methylene
                       1.32           1 alpha -N
                       0.32           1 beta -N=C-1:C*C*C*C*C*C*1
H    8.11              8.11        benzylidenimin
                       0.00           1 -C
```

©IJRASET (UGC Approved Journal): All Rights are Reserved

1) *Compound 3:*




$N^1$-(3-nitrobenzylidene)ethane-1,2-diamine

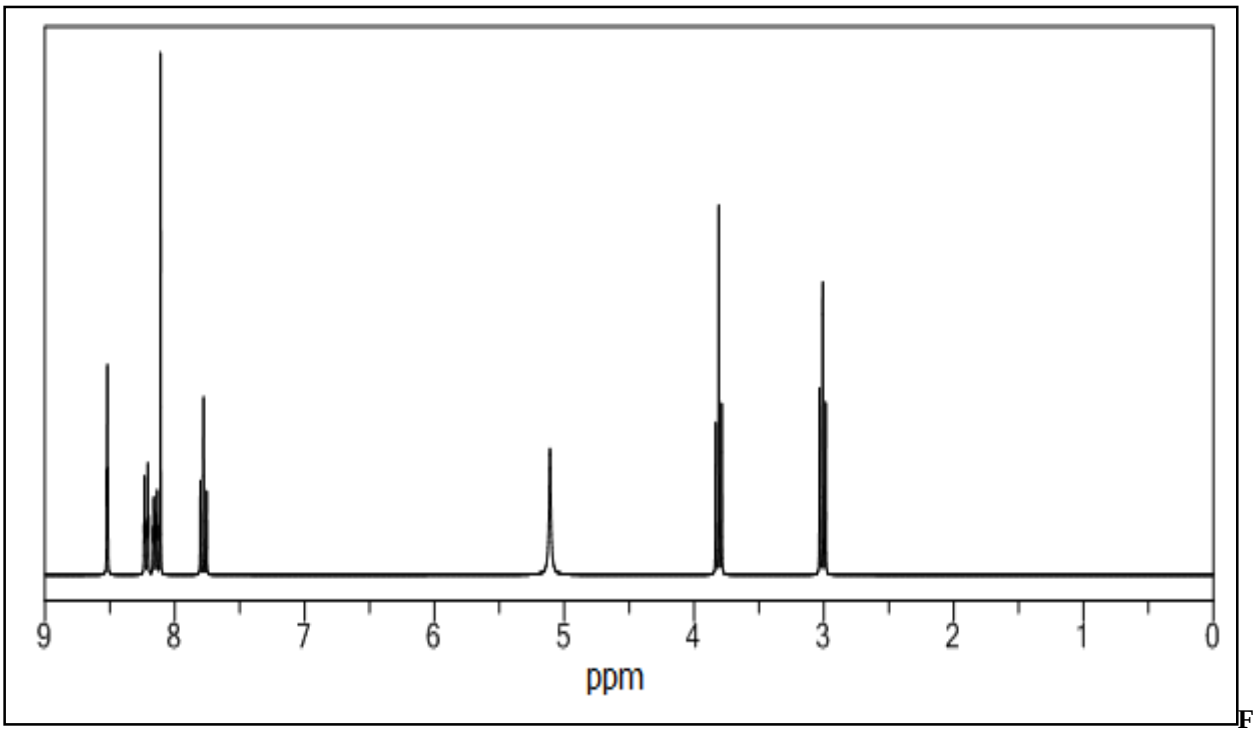



Figure 2.3: $^1$HNMR spectra of $N^1$-(3-nitrobenzylidene)ethane-1,2-diamine.

I. *Result and discussio*

Total number of protons= 11

-$NH_2$= 5.11 ppm

-$CH_2$= 3.01 ppm

-$CH_2$= 3.81 ppm

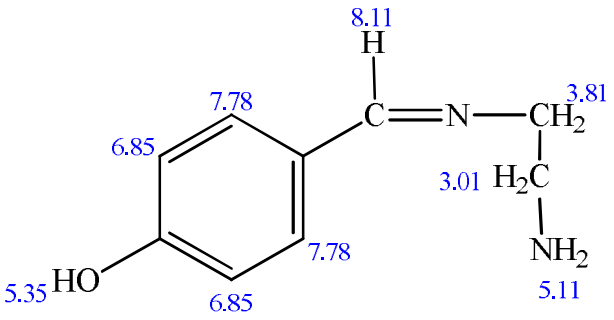
-CH= 8.11 ppm

-Aromatic protons= 8.52, 8.15, 8.22, 7.78 ppm.

©IJRASET (UGC Approved Journal): All Rights are Reserved

| Node | Shift | Base + Inc. | Comment (ppm rel. to TMS) |
|---|---|---|---|
| NH2 | 5.11 | 2.00 | amine |
| | | 3.11 | general corrections |
| CH | 8.15 | 7.29 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | 0.93 | 1 -N(=O)=O from 1-benzene |
| | | -0.07 | general corrections |
| CH | 8.52 | 7.62 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | 0.93 | 1 -N(=O)=O from 1-benzene |
| | | -0.03 | general corrections |
| CH | 8.22 | 7.62 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | 0.39 | 1 -N(=O)=O from 1-benzene |
| | | 0.21 | general corrections |
| CH | 7.78 | 7.29 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | 0.26 | 1 -N(=O)=O from 1-benzene |
| | | 0.23 | general corrections |
| CH2 | 3.81 | 1.37 | methylene |
| | | 2.22 | 1 alpha -N=C-1:C*C*C*C*C*C*1 |
| | | 0.22 | 1 beta -N |
| CH2 | 3.01 | 1.37 | methylene |
| | | 1.32 | 1 alpha -N |
| | | 0.32 | 1 beta -N=C-1:C*C*C*C*C*C*1 |
| H | 8.11 | 8.11 | benzylidenimin |
| | | 0.00 | 1 -C |

*1) Compound4*




4-(((2-aminoethyl)imino)methyl)phenol

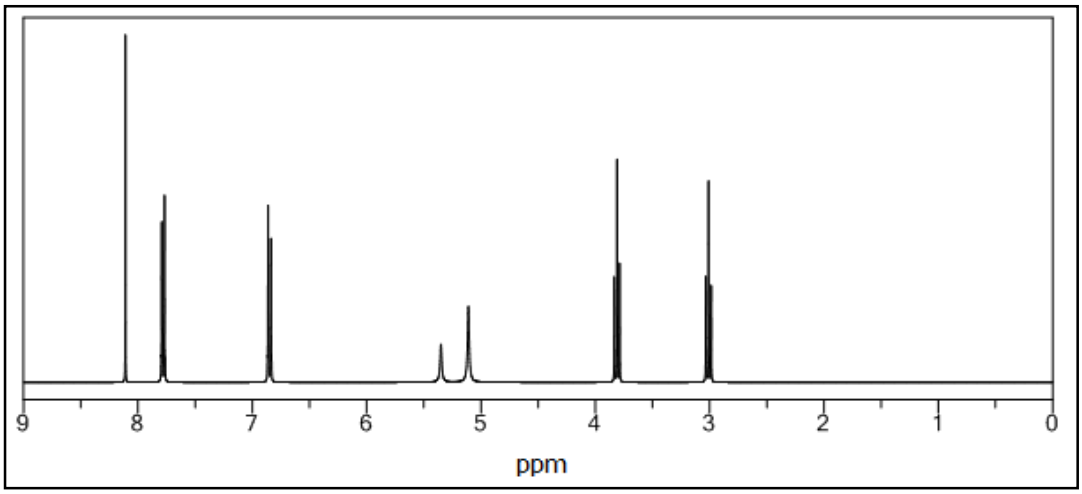



Figure 2.4: [1]HNMR spectra of 4-(((2-aminoethyl)imino)methyl)phenol.

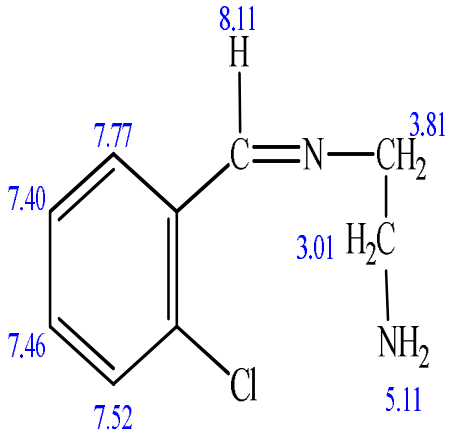
©IJRASET (UGC Approved Journal): All Rights are Reserved

*J. Result and discussion:*

Total number of protons= 12
-NH2= 5.11 ppm
-CH2= 3.01 ppm
-CH2=3.81 ppm
-CH= 8.11 ppm
-OH= 5.35 ppm
-Aromatic protons= 6.85, 6.85, 7.78, 7.78 ppm.

| Node | Shift | Base + Inc. | Comment (ppm rel. to TMS) |
|---|---|---|---|
| OH | 5.35 | 5.00 | aromatic C-OH |
| | | 0.35 | general corrections |
| NH2 | 5.11 | 2.00 | amine |
| | | 3.11 | general corrections |
| CH | 6.85 | 7.29 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | -0.53 | 1 -O from 1-benzene |
| | | 0.09 | general corrections |
| CH | 7.78 | 7.62 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | -0.17 | 1 -O from 1-benzene |
| | | 0.33 | general corrections |
| CH | 6.85 | 7.29 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | -0.53 | 1 -O from 1-benzene |
| | | 0.09 | general corrections |
| CH | 7.78 | 7.62 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | -0.17 | 1 -O from 1-benzene |
| | | 0.33 | general corrections |
| CH2 | 3.81 | 1.37 | methylene |
| | | 2.22 | 1 alpha -N=C-1:C*C*C*C*C*C*1 |
| | | 0.22 | 1 beta -N |
| CH2 | 3.01 | 1.37 | methylene |
| | | 1.32 | 1 alpha -N |
| | | 0.32 | 1 beta -N=C-1:C*C*C*C*C*C*1 |
| H | 8.11 | 8.11 | benzylidenimin |
| | | 0.00 | 1 -C |

*1) Compound 5*




$N^1$-(2-chlorobenzylidene)ethane-1,2-diamine

©IJRASET (UGC Approved Journal): All Rights are Reserved

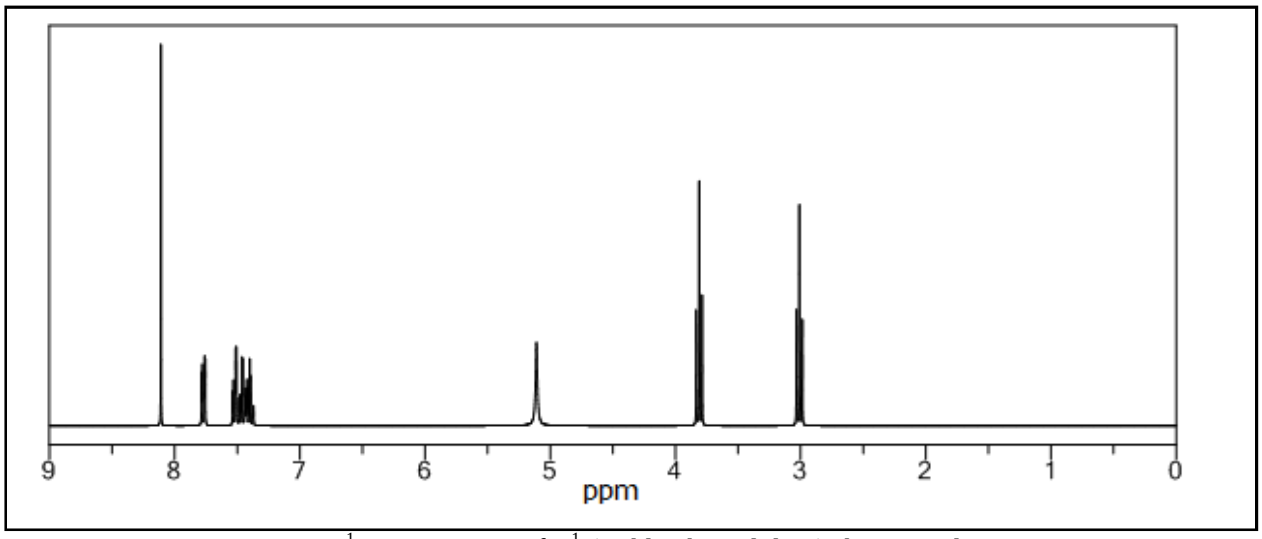



Figure 2.5: $^{1}$HNMR spectra of N$^{1}$-(2-chlorobenzylidene)ethane-1,2-diamine.

*K. Result and discussion*

Total number of protons= 11

-$NH_2$= 5.11 ppm

-$CH_2$= 3.01 ppm

-$CH_2$= 3.81 ppm

-CH=8.11 ppm

-Aromatic protons= 7.52, 7.46, 7.40, 7.77 ppm.

| Node | Shift | Base + Inc. | Comment (ppm rel. to TMS) |
|---|---|---|---|
| NH2 | 5.11 | 2.00 | amine |
| | | 3.11 | general corrections |
| CH | 7.52 | 7.29 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | 0.01 | 1 -Cl from 1-benzene |
| | | 0.22 | general corrections |
| CH | 7.77 | 7.62 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | -0.06 | 1 -Cl from 1-benzene |
| | | 0.21 | general corrections |
| CH | 7.46 | 7.29 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | -0.06 | 1 -Cl from 1-benzene |
| | | 0.23 | general corrections |
| CH | 7.40 | 7.29 | benzylidenimin |
| | | 0.00 | 1 -C |
| | | -0.12 | 1 -Cl from 1-benzene |
| | | 0.23 | general corrections |
| CH2 | 3.81 | 1.37 | methylene |
| | | 2.22 | 1 alpha -N=C-1:C*C*C*C*C*C*1 |
| | | 0.22 | 1 beta -N |
| CH2 | 3.01 | 1.37 | methylene |
| | | 1.32 | 1 alpha -N |
| | | 0.32 | 1 beta -N=C-1:C*C*C*C*C*C*1 |
| H | 8.11 | 8.11 | benzylidenimin |
| | | 0.00 | 1 -C |

©IJRASET (UGC Approved Journal): All Rights are Reserved

*ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor:6.887*
*Volume 5 Issue XI November 2017- Available at www.ijraset.com*

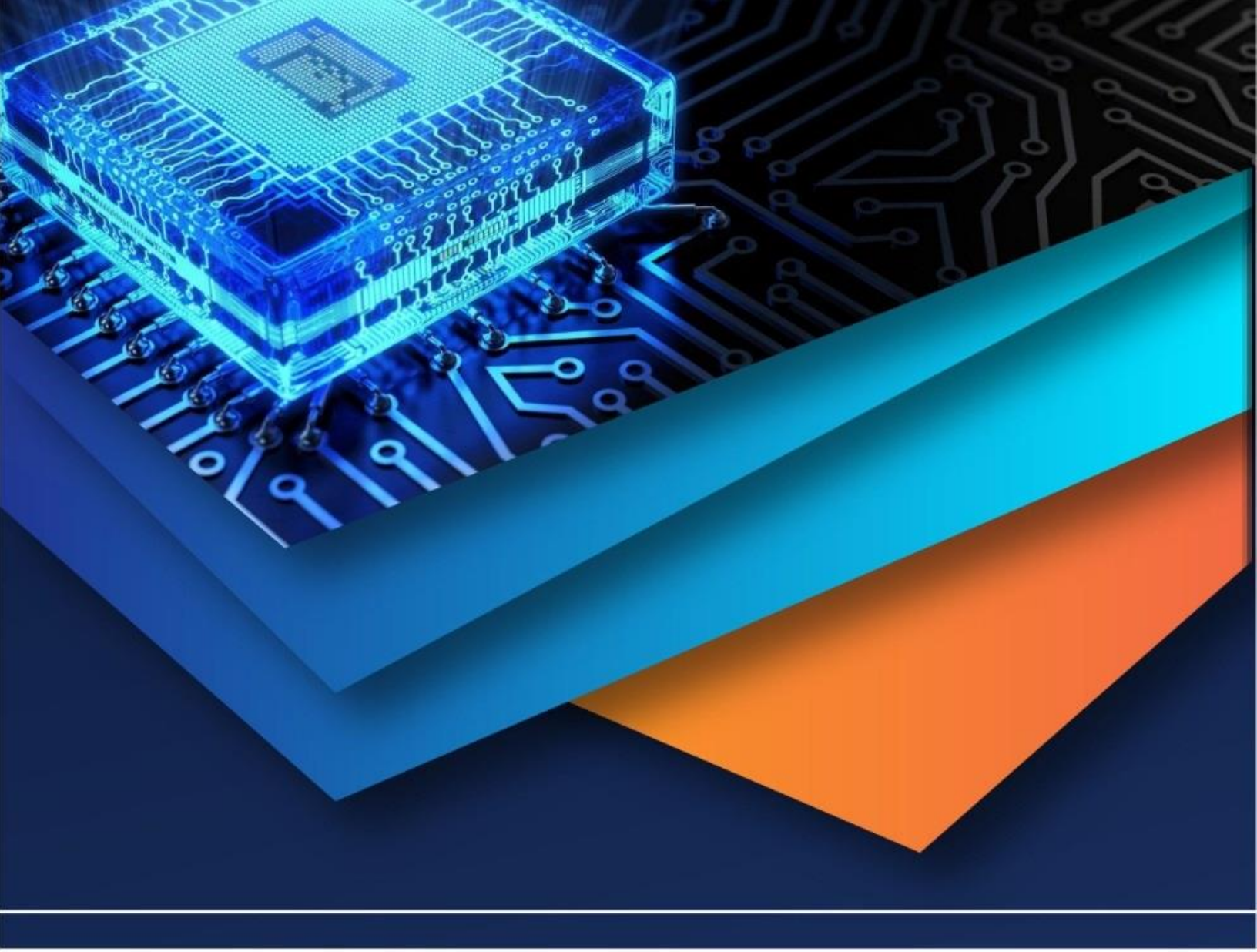
## III. CONCLUSION

The present study demonstrates a more convenient, efficient and eco friendly method of preparation of Schiff's base derivatives from various substituted aromatic aldehydes and ethylene diammine under mild conditions using water as a green solvent at a constant temperature. Thus making it an efficient green synthetic procedure.

## REFERENCES

[1] V. K. Ahluwalia and M. Kidwai, New Trends in Green Chem., Anamaya Publisher, New Delhi, 2004
[2] W. Zhang and B. W. Cue, A text book on Green Techniques for Organic Synthesis and Medicinal Chemistry, ISBN-2012
[3] C. J. Li, Chemical Reviews, 2005, 105:3095
[4] G. W. V. Cave, C. L. Raston and J. L. Scott, Chemical Communications., 2001, 21:2159-216
[5] P. T. Anastas and J. C. Warner, Oxford Science Publications, Oxford, 1998
[6] P. Singh, R. L. Goel and B.P. Singh, Journal of the Indian Chemical Society, 1975, 52:958
[7] Z. Cimerman, S. Miljanic and N. Galic, Crotica Chemical Acta., 2000, 73(1):81-95
[8] M. N. Ibrahim, K. J. Hamad and S.H. Al-Joroshi, Asian Journal of Chemistry, 2006, 18(3):2404-2406.

©IJRASET (UGC Approved Journal): All Rights are Reserved



10.22214/IJRASET

45.98

IMPACT FACTOR: 7.129

IMPACT FACTOR: 7.429

# INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Call : 08813907089 (24*7 Support on Whatsapp)