iJRASET
International Journal For Research in Applied Science and Engineering Technology



INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 10 **Issue:** II **Month of publication:** February 2022

DOI: https://doi.org/10.22214/ijraset.2022.40379

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: 7.538
Volume 10 Issue II Feb 2022- Available at www.ijraset.com

Lemon Juice Catalysed Green Synthesis of Triazole Based Schiff Base, Its Physico-Chemical & Spectral Characterisation and Determination of its Binding Stoichiometry with Cu^{2+} ion

Vivek Kumar[1], Abhay Kumar[2], Rajesh Kumar[3]
[1]*Department of Chemistry, Patna University, Patna, Bihar, India*
[2]*Department of Chemistry, Patna University, Patna, Bihar, India*
[3]*Department of Chemistry, Patna University, Patna, Bihar, India*

Abstract: ***In the recent years Environmental Pollution has emerged as a major challenge for human society. Since the start of Industrial Revolution in England, the amount of chemical waste and toxic substances are increasing exponentially in the environment. The traditional synthesis of chemical substances is accompanied by use of large amount of energy, harmful solvents and release of toxic substances in environment. The green synthesis aims to minimise the release and use of harmful toxic material in synthesis. In the present paper we have prepared a triazole derivative, 4-[(E)-Benzylideneamino]-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione through green synthetic route. The reaction was catalysed by lemon juice and it was carried out in solvent-free condition through mechano-chemical stirring method. The physico-chemical properties were studied. The FT-IR spectral analysis confirmed the structure of Schiff base. The stoichiometry of binding with Cu^{2+} was determined through Method of Continuous Variation. The Schiff base- metal ratio was found to be 1:1.***

Keywords: Green Synthesis, Mechano-Chemical Stirring Method, Triazole, Schiff Base, Lemon Juice, Physico-Chemical Properties, Job's Plot, Stoichiometry of Binding.

I. INTRODUCTION

Green chemistry involves elimination of use and generation of hazardous material in production of chemical products and methods [1]. Economic goal of the human society can be achieved through green methods without damaging environment [2]. The organic solvents which are used in synthesis of various chemical compounds have adverse effect on environment [3]. Green solvents are biodegradable and obtained from natural occurring product [4,5]. In 1864, Schiff base was prepared by Hugo Schiff by condensation of an aldehyde and amine [6]. Schiff base are chemical compounds with general formula $R_1R_2C=NR$ ($R \neq H$) [7]. Schiff bases are easy to synthesize and show variety of biological activity like Antioxidant [8], Anti-Inflammatory [9], Anti-Microbial, Antifungal, Antiviral, Synergistic action on Insecticides, Plant growth Regulator, Allergy inhibitor, Analgesic [10]. The naturally occurring fruit juices have been used as biocatalyst in organic synthesis recently [11]. Lemon juice. Grape juice, Extract of raw Mango have been used to synthesize Schiff base [12,13,14]. In the present research, a Triazole derived Schiff base have been prepared through Green synthetic method using Lemon juice as catalyst.

II. EXPERIMENTAL

A. *Materials and Methods*

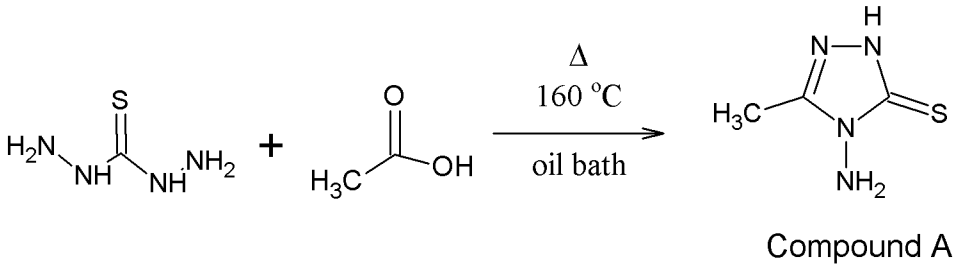
All the chemicals used were either of Merck, Finar, Nice, BDH or Loba. The melting point was determined by open-capillary method. The molar mass was calculated using Rast Method using Camphor as solvent [15]. The metal binding property of the Schiff base was studied using Method of Continuous Variation [16]. The absorbance value were taken using Systronic Photoelectric Colorimeter 112. The FT-IR Spectra was recorded by Agilent Cary 630 FTIR instrument in the range 4000-450 cm^{-1}.

B. *Synthesis of Schiff base*

1) *Preparation of Lemon Juice :* The lemons were bought from local market. They were pressed manually and juice was filtered using filter paper to obtain clear lemon juice.
2) *Synthesis of 4-amino-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione :* The compound was synthesized by reported method [17] and recrystallised using ethanol.

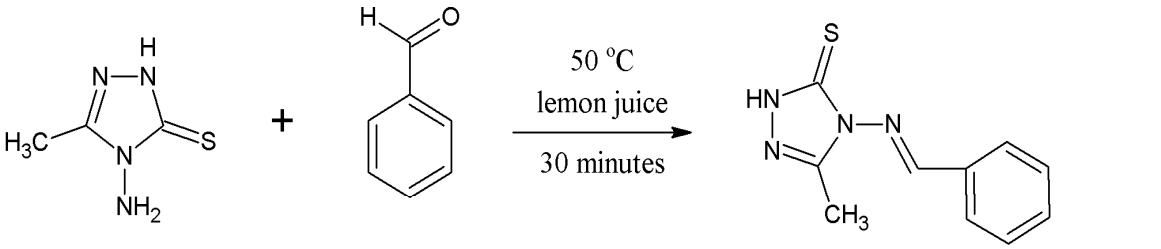
©IJRASET: All Rights are Reserved

ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.538
Volume 10 Issue II Feb 2022- Available at www.ijraset.com



Compound A

3) *Green Synthesis of Schiff Base :* 0.002 mole of compound A and 0.002 mole of Benzaldehyde was taken in a beaker. 0.5 ml of lemon juice was added. The reaction mixture was stirred for 30 minutes at 50°C on hot plate. The reaction mixture colour turns yellow. 25ml of distilled water was added and product was filtered, washed with distilled water and recrystallised with ethanol. The yield of the product was 71.3 %.



4-[(*E*)-benzylideneamino]-5-methyl-2
,4-dihydro-3*H*-1,2,4-triazole-3-thione



Fig. 1 Schiff Base , 4-[(E)-Benzylideneamino]-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione Recrystallised from Ethanol.

©IJRASET: All Rights are Reserved

ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.538
Volume 10 Issue II Feb 2022- Available at www.ijraset.com

III.RESULTS AND DISCUSSION

A. *Physico-Chemical Analysis*

The yield of the synthesized Triazole based Schiff Base through green synthetic route is 71.3%. It was recrystallised using minimum amount of ethanol. Pale-Yellow coloured crystalline solid was obtained. The prepared compound was stable in air. The melting point of the synthesized compound was found 195-197°C. The value of molar mass is 220.56 which was calculated using Rast Method. Physico-Chemical properties of the Schiff Base is given in Table I.

Table I
Physico-Chemical Properties Of Synthesized Schiff Base

SL. No.	PROPERTIES	VALUE
1.	IUPAC Name	4-[(E)-Benzylideneamino]-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione
2.	Colour	Pale-Yellow
3.	Physical State	Crystalline Solid
4.	Yield	71.3 %
5.	Melting Point	195-197°C
6.	Stability	Stable in Air
7.	Recrystallisation Solvent	Ethanol
8.	Molar Mass	220.56

The solubility of the synthesized Schiff Base was checked in various organic solvents. The Triazole based Schiff Base is completely soluble in DMSO, partially soluble in ethanol, chloroform and insoluble in acetone and water. The solubility of the compound in various organic solvents is given in Table II.

Table II
Solubility Data of Synthesized Schiff Base

SL No.	SOLVENT	SOLUBILITY
1.	WATER	INSOLUBLE
2.	ETHANOL	PARTIALLY SOLUBLE
3.	ACETONE	INSOLUBLE
4.	CHLOROFORM	PARTIALLY SOLUBLE
5.	DMSO	SOLUBLE

B. *FT-IR Spectral Analysis*

The FT-IR spectra of the Triazole based Schiff Base was recorded using Agilent Cary 630 FTIR instrument in the frequency range 4000-450cm^{-1}. The IR spectral frequency bands of the compound is mentioned in Table III.

Table III
FT-IR Spectral Band Positions of Synthesized Schiff Base

WAVENUMBER (in cm^{-1})	ASSIGNMENT
3392.52, 3284.55	ν (NH) azole ring
755.35	N-H Wagging
2986.92	ν_{as} (CH_3)
2925.59	ν_{s} (CH_3)
1447.94	δ_{as} (CH_3)
3159.45	ν (C=H alkene)

©IJRASET: All Rights are Reserved | SJ Impact Factor 7.538 | ISRA Journal Impact Factor 7.894 |

691.23	Out of plane bending of ring C-H
1301.16	In plane bending of ring C-H
1527.85	Ring stretch phenyl
1244.52	ν (C-N)
1602.87	ν (C=N)
1065.48	ν (C=S)
1385.20	Ring stretch azole

ν = stretching ν_{as} = asymmetrical stretching ν_s = symmetrical stretching δ_{as} = asymmetrical bending

The IR spectrum of the Schiff Base, 4-[(E)-Benzylideneamino]-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione shows stretches between 3392.52 cm^{-1} and 2925.59 cm^{-1}. These are located at 3392.52cm^{-1}, 3284.55cm^{-1}, 3159.45cm^{-1},2986.92cm^{-1}, 2925.59cm^{-1} and belongs to NH and CH_3 stretches. The compound contain two aromatic ring i.e. phenyl and azole ring which shows ring stretches at 1527.85 cm^{-1} and 1385.20 cm^{-1} respectively. The presence of thiocarbonyl group is confirmed by stretching band at 1065.48cm^{-1}. The formation of Schiff base is confirmed by C=N stretch at 1602.87 cm^{-1}.

C. Determination of stoichiometry of binding of synthesized Schiff Base with Cu^{2+} ion

The binding stoichiometry of 4-[(E)-Benzylideneamino]-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (ligand) with Cu^{2+} ion was determined by Method of Continuous Variation. The absorbance data was recorded on Systronic Photoelectric Colorimeter 112 at the wavelength 720nm. Two stock solutions were prepared. 100 ml of 0.001M ligand solution was obtained by dissolving 0.022g of Schiff base in ethanol. Similarly, 100 ml of 0.001M Cu^{2+} was obtained by dissolving 0.017g of $CuCl_2.2H_2O$ in ethanol. Eleven solutions of 10 ml each were made by mixing ligand solution and salt solution such that different mole-fractions of ligand is obtained while overall volume remains the same. The absorbance value for each solution was collected. The absorbance was plotted against mole fraction to generate a Job's Plot. The Job's Plot is shown in Fig. 2

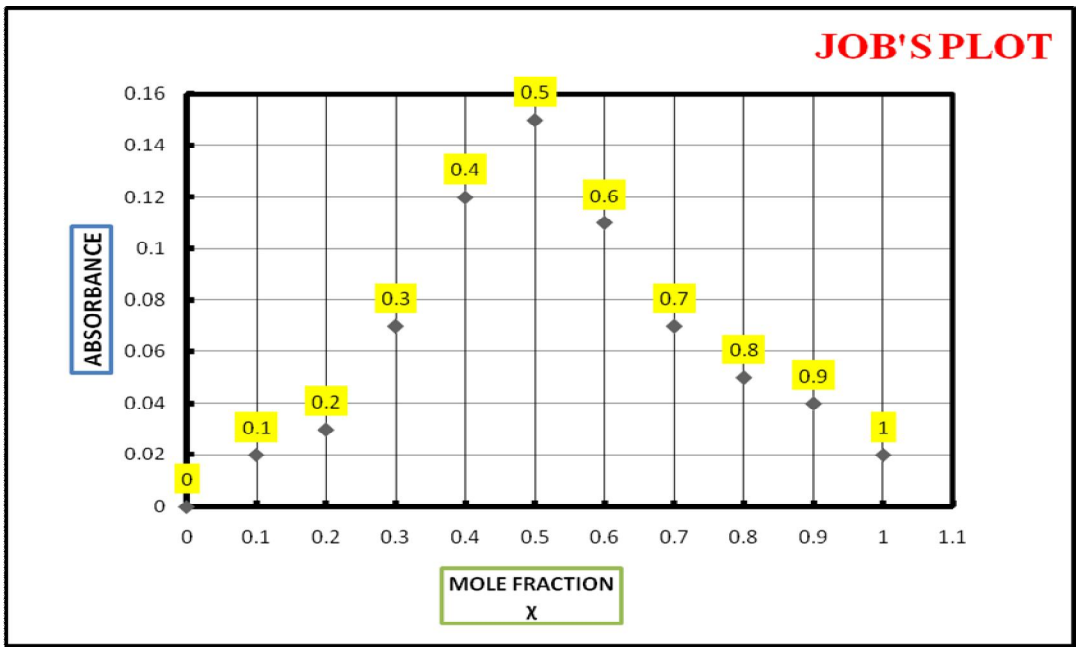


Fig. 2 Job's Plot of Cu^{2+} with ligand at λ_{abs} = 720nm

The analysis of Job's Plot revealed that slope changes from positive to negative at mole-fraction 0.5 which is called turning point. The turning point at 0.5 indicates the formation of 1:1 complex. Hence, the binding stoichiometry between Cu^{2+} and ligand is 1:1.

IV. CONCLUSIONS

In summary we have synthesized a Triazole based Schiff base through green synthetic method. The synthesis was accomplished using mechano-chemical stirring and lemon juice was used as catalyst. The synthesized Schiff Base was characterized by physico-chemical properties and FT-IR Spectra. The stoichiometry of binding of Schiff base with Cu^{2+} was determined using the Method of Continuous Variation. The analysis of Job's Plot indicates the formation of 1:1 complex.

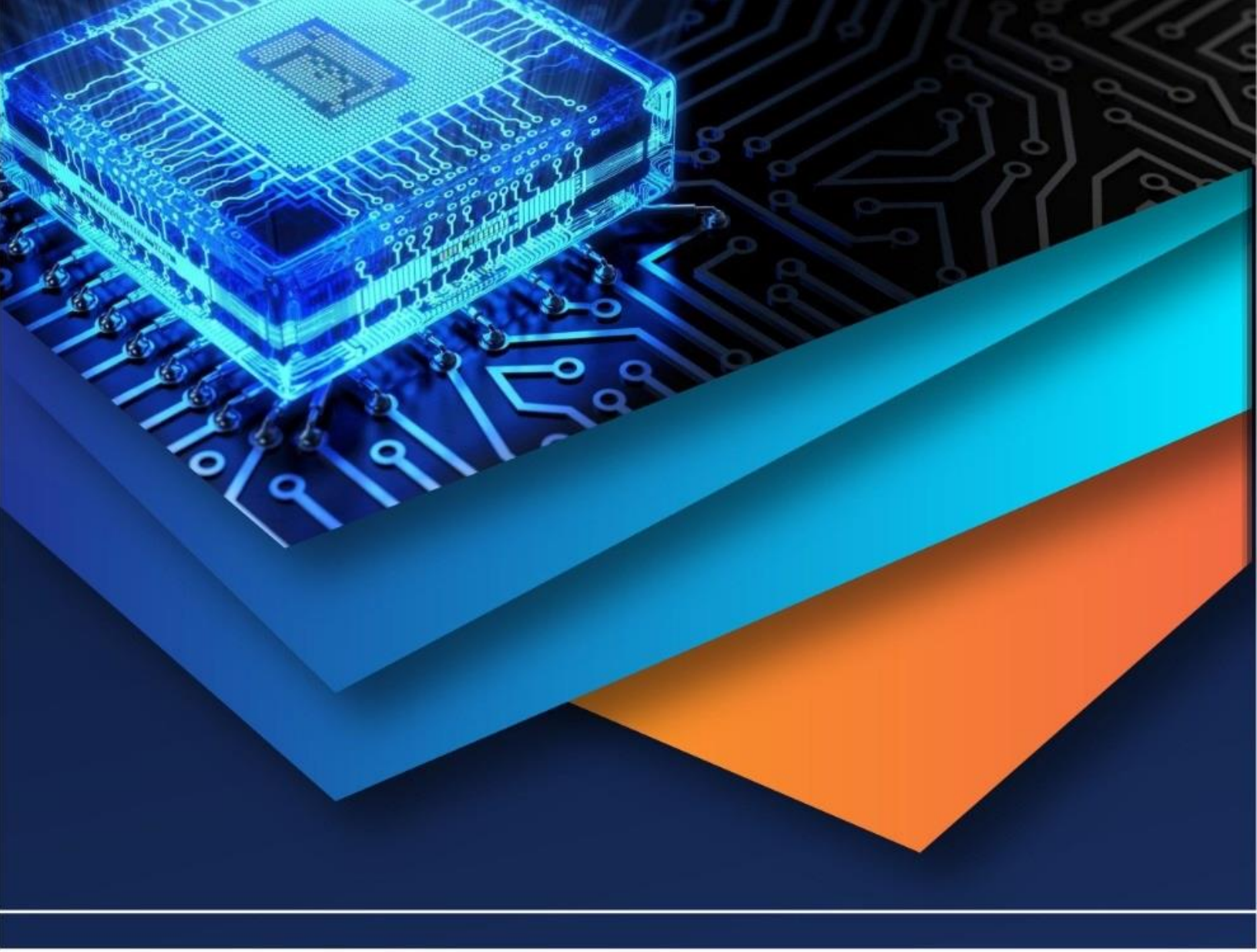
V. ACKNOWLEDGMENT

Vivek Kumar wishes to acknowledge CSIR-HRDG, New Delhi for providing financial support to carry out the research work under CSIR-NET-JRF Scheme (File No.- 09/138(0078)/2019-EMR-I). We also want to acknowledge The Head, DST-SAIF, CDRI Lucknow for recording FT-IR Spectra of the compound.

REFERENCES

[1] Anastas, Paul T., and Tracy C. Williamson, eds. Green chemistry: designing chemistry for the environment. American Chemical Society, 1996.
[2] Anastas P, Eghbali N. Green chemistry: principles and practice. Chemical Society Reviews. 2010;39(1):301-12.
[3] Cseri L, Razali M, Pogany P, Szekely G. Organic solvents in sustainable synthesis and engineering. InGreen Chemistry 2018 Jan 1 (pp. 513-553). Elsevier.
[4] Clark JH. Green chemistry: challenges and opportunities. Green Chemistry. 1999;1(1):1-8.
[5] Zhang, Wei, and Berkeley W. Cue, eds. Green techniques for organic synthesis and medicinal chemistry. John Wiley & Sons, 2018.
[6] Cozzi PG. Metal–Salen Schiff base complexes in catalysis: practical aspects. Chemical Society Reviews. 2004;33(7):410-21.
[7] IUPAC. Compendium of Chemical Terminology, 2nd ed. (the "Gold Book"). Compiled by A. D. McNaught and A. Wilkinson. Blackwell Scientific Publications, Oxford (1997). Online version (2019-) created by S. J. Chalk. ISBN 0-9678550-9-8. https://doi.org/10.1351/goldbook.
[8] Shah SS, Shah D, Khan I, Ahmad S, Ali U, ur Rahman A. Synthesis and Antioxidant Activities of Schiff Bases and Their Complexes: An Updated Review.
[9] Parashar RK, Sharma RC, Kumar A, Mohan G. Stability studies in relation to IR data of some schiff base complexes of transition metals and their biological and pharmacological studies. Inorganica chimica acta. 1988 Mar 1;151(3):201-8.
[10] Kumar, S., Dhar, D.N. and Saxena, P.N., 2009. Applications of metal complexes of Schiff bases-A review.
[11] Gulati, Susheel, and Rajvir Singh. "Green and Environmentally Benign Organic Synthesis by Using Fruit Juice as Biocatalyst: A Review." International Research Journal of Pure and Applied Chemistry (2018): 1-15.
[12] Alikhani A, Foroughifar N, Pasdar H. Lemon juice as a natural catalyse for synthesis of Shiff's base: a green chemistry approach. International Journal Of Advanced Engineering Research And Science. 2018;5(2):237378.
[13] Yadav G, Mani JV. Green synthesis of Schiff bases by using natural acid catalysts. International Journal of Science and Research. 2015;4(2):121-7.
[14] Patil S, Jadhav SD, Patil UP. Natural acid catalyzed synthesis of Schiff base under solvent-free condition: as a green approach. Archives of Applied Science Research. 2012;4(2):1074-8.
[15] Wawzonek S. A modified Rast method for molecular weights. Journal of Chemical Education. 1972 Jun;49(6):399.
[16] Wilkinson SM, Sheedy TM, New EJ. Synthesis and characterization of metal complexes with schiff base ligands. Journal of Chemical Education. 2016 Feb 9;93(2):351-4.
[17] Nikpour M, Motamedi H. Convenient Access to 1, 3-Dimethyl [1, 2, 4] Triazolo [3, 4-b][1, 3, 4]-Thiadiazol-1-Ium and 7H-[1, 2, 4] Triazolo [4, 3-b][1, 2, 4] Triazol-1-Ium Salts. Chemistry of Heterocyclic Compounds. 2015 Feb;51(2):159-61.

.



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)