iJRASET
International Journal For Research in Applied Science and Engineering Technology



INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 11 **Issue:** XII **Month of publication:** December 2023

DOI: https://doi.org/10.22214/ijraset.2023.57829

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 11 Issue XII Dec 2023- Available at www.ijraset.com

Synthesis, Characterization and Antimicrobial Study of Some Transition Metal Complexes with Aldimine

Dinkar P. Kotwal
Department of Chemistry, Dnyanopasak Shikshan Mandal's Arts, Commerce and Science College, Jintur-431509, MS, India

Abstract: Aldimine ligand 2-Hydroxy-4,5- diiodo-benzylidene-4-hydroxy-aniline (L) and its complexes with Cu (II), Ni (II), Co (II) and Zn (II) were prepared and characterized by analytical, spectroscopic (IR, UV-Vis) techniques, thermal study, electrical conductivity and magnetic measurements. The results indicate that the ligand coordinate through azomithine nitrogen and oxygen of enolic group. The complexes were further screened for antimicrobial activity.
Keywords: Aldimine, Transition metal, metal complexes, spectral study, antimicrobial study.

I. INTRODUCTION

Aldimine are important class of ligand in coordination chemistry and their complex formation ability containing different donor atom is widely reported. The chemistry of transition metal complexes containing heterocyclic donor continues to be of interest on account of their biological importance[1]. The aldimine metal complexes show antimicrobial[2], fungicidal[3] anti-cancer[4] and anti-inflammatory activity[5].Thus the present study describe synthesis, characterization and antimicrobial studies of some aldimine transition metal complexes.

A. Experimental

The ligand 2-Hydroxy-4,5- diiodo-benzylidene-4-hydroxy-aniline (L) has been characterized by infrared spectral studies. The complex characterized by infrared spectral studies, electronic spectral studies, magnetic moment, thermal analysis and conductivity measurement. The antimicrobial activity of the investigated compound was tested by the paper diffusion method.

B. Synthesis of Aldimine

The aldimine was synthesized by reported procedure[6]. To the solution of 2-hydroxy-4, 5-diiodobenzyldehyde 0.373 gm (0.001 mole) in 25 ml ethanol and 0.109 gm (0.001 mol) p-amino phenol in 25 ml ethanol was added. The solution was refluxed for 5-6 hrs. The solution was monitored on TLC, on completion of reaction the solution was concentrated, cooled and poured in water the separated solid was filtered, washed with water and recrystallized from ethyl alcohol and dried over calcium chloride in vacuum. The composition and yield of aldimine have shown in table no 1.

C. Synthesis of Metal Complexes

The metal complexes of Cu (II), Ni (II), Co (II) and Zn (II) with ligand were prepared by refluxing the ethanolic solution of metal nitrate and ligand in 1:2 molar ratios. The pH of solution was adjusted by alcoholic ammonia.
Ligand (0.02 mole) in slight excess was taken in a round bottom flask containing 30 ml anhydrous ethanol and refluxed for few minutes with constant stirring to ensure complete dissolution; a solution of the appropriate metal nitrate (0.01 M) in 20 ml of anhydrous ethanol was then added drop by drop with constant-stirring in the hot solution of ligand. The content was refluxed five hours. After cooling, the pH of the mixture was maintained by the addition of alcoholic ammonia solution. The resultant precipitate was digested for one hour. The precipitate was filtered, washed with hot ethanol and dried in vacuum desiccators over anhydrous granular calcium chloride. The composition and yield of metal complexes have shown in table no 1.

II. RESULT AND DISCUSSION

The metal complexes are colored and stable to air and moisture. They are insoluble in water, sparingly soluble in benzene, chloroform, carbon tetrachloride etc. and completely soluble in dimethyl formamide (DMF) and dimethyl sulfoxide (DMSO). Elemental analysis suggests that the complex have 1:2(metal-ligand) stoichiometry.

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

The conductivity value in DMF (1 x 10^{-3}M) reveals their electrolyte nature [7,8]. Based on elementary chemical analysis the formula, $ML_2(H_2O)_2(NO_3)_2$ were suggested for all compounds. In IR spectrum of ligand, shows band at 2823.60 cm^{-1} assigned to –OH stretching, the lowering of normal free –OH stretching frequency from 3600- 3500 cm^{-1} to above value is expected due to the strong intramolecular hydrogen bonding.[9] A bond at 1620.10 cm^{-1} assigned to azomethine (C=N) stretching vibration. A bond at 1274.90 cm^{-1} is assigned to enolic C-O stretching frequency.

Table 1: Analytical data of ligand and metal complexes

Compound	Colour	Yield (%)	Formula	Mol Wt.	% Analysis Found(Calculated)			
					C	H	M	N
Ligand(L)	Dark Brown	64	$C_{13}H_9I_2NO_2$	465.03	33.45 (33.58)	1.86 (1.95)	--	2.92 (3.01)
$(L)_2Cu(H_2O)_2(NO_3)_2$	Black	59	$C_{26}H_{22}I_4CuN_2O_6$	1029.63	30.26 (30.33)	2.06 (2.15)	5.90 (6.17)	2.65 (2.72)
$(L)_2Ni(H_2O)_2(NO_3)_2$	Brown	62	$C_{26}H_{22}I_4Ni\ N_2O_6$	1024.77	31.37 (30.47)	2.11 (2.16)	5.51 (5.73)	2.68 (.73)
$(L)_2Co(H_2O)_2(NO_3)_2$	Brown	73	$C_{26}H_{22}I_4CoN_2O_6$	1025.01	30.40 (30.47)	2.07 (2.16)	541 (5.75)	2.67 (2.73)
$(L)_2Zn(H_2O)_2(NO_3)_2$	Pink	74	$C_{26}H_{22}I_4ZnN_2O_6$	1031.47	30.20 (30.28)	2.09 (2.15)	6.02 (6.34)	2.67 (2.72)

In the spectra of Cu(II) complexes the band at 2823.60 cm^{-1} disappears. The band at 1620.10 cm^{-1} is shifts to lower frequency (1615.10 cm^{-1}), indicating the co-ordination of azomethine nitrogen to metal atom. In spectra of complex the broad band at 3354.73 cm^{-1}, indicating the presence of coordinated water. The bands at 1274.90 cm^{-1} show upward shift (1285.90 cm^{-1}) suggest the bonding of enolic oxygen of aldimine base with metal during complexation[10,11] , further confirmed by a new band. The new bands at 527.18 cm^{-1} and 498.77 cm^{-1} in the spectra of complex are assigned to stretching vibration of M-N and M-O bands respectively[12].

Table-2: IR frequency (cm^{-1}) of ligand and complexes

Ligand / Complex	ν (OH) Water	ν (OH) Phenoic	ν (C-O)	ν (C=N)	ν (M-N)	ν (M-O)
L	-	2823.60	1274.90	1620.10	-	-
L-Cu	3354.73	-	1285.90	1615.10	527.18	498.77

The UV-Visible spectra of copper complexes of ligand exhibit bands at 15576 cm^{-1}, 27932 cm^{-1} attributed to $^3Eg \rightarrow {}^2T_{2g}$ and charge transfer transition which indicate distorted octahedral geometry of Cu (II) complexes, which further supported by μ_{eff} value 1.90 B.M.[13].

The ligand field parameter values (Dq and LFSE) observed at 15576 cm^{-1} and 44.47 kcal $mole^{-1}$ respectively and these are in good agreement with the distorted octahedral Cu (II) complexes.

The electronic spectra of Ni (II) complex exhibit bands in the three regions as 9587 cm^{-1} 15948 cm^{-1} and 24213 cm^{-1}. This pattern of absorption may be assigned to transition $^3A_{2g} \rightarrow {}^3T_{2g}(F)$, $^3A_{2g} \rightarrow {}^3T_{1g}$ (F) and $^3A_{2g} \rightarrow T_{1g}$ (P) respectively. This may be assigned to characteristic three spin allowed transition of octahedral complexes[14], further supported by μeff value 3.39 B.M.

The reduction of B values for the Ni(II) complexes (760.00 cm^{-1}) as compared to the free ion value for Ni(II) ion (1030 cm^{-1}) reveals complex formation. The Nephelauxetic ratio (β) obtained are less than unity (0.737) in this case suggesting considerable amount of covalent character of the metal ligand bonds. The LFSE value (27.37 Kcal $mole^{-1}$) indicate considerable stability of Ni(II) complexes. The energy ratio ν_2/ν_1 (1.663) being close to reported values (1.6-1.82) as well as the 10Dq (9587cm^{-1}) and percentage of β found (35.68) indicate that the Ni (II) complex is in octahedral geometry.

The electronic spectra of Co (II) complex exhibit bonds at 9451 cm^{-1} , 18281 cm^{-1} and 22123 cm^{-1}. These bands may be assigned to to $^4T_{1g}(F) \rightarrow {}^4T_{2g}$, $^4T_{1g}(F) \rightarrow {}^4A_{2g}$, (F) and $4T_{1g}(F) \rightarrow {}^4T_{1g}$, (p) respectively, suggesting a high spin octahedral geometry around the cobalt ion. Octahedral geometry around cobalt ion[15] further supported by μ_{eff} value 4.73B.M.

The reduction in B values for all the Co(II) complexes (803.40 cm^{-1}) as compared to the free ion value for Co(II) ion (971 cm^{-1}) reveals complexes formation thereby favoring the orbital overlap and delocalization of d orbital. The Nephelauxetic ratio (β) in each case is less than unity (0.827) and their %β values (20.91 %) indicate partial covalent character in metal ligand bond. The LFSE value (25.21 Kcal $mole^{-1}$) indicates considerable stability of Co (II) complexes.

The electronic spectra of Zn (II) complex exhibits bands at 27777 cm^{-1} attributed to charge transfer transition suggesting an octahedral environment[16]. This complex is diamagnetic in nature.

The simultaneous use of TG and DTA is made in the present study of metal complexes with a view to understand stoichiometry, thermal stability, the presence and nature of water molecules. The water in inorganic compounds may be classified as lattice water and coordinated water. There is however, no definite border line between the two. The former term denotes water molecules trapped in the crystalline lattice, either by weak bonds to the anion or by weak ionic bonds to the metal or by both, where as the latter denotes water molecules bonded to the metal through partial covalent bonds or coordinate bonds. According to Freman and Carrol[18] the water eliminated below 150 °C can be considered as lattice water and above 150 °C as water coordinated to metal ion[19].

A close look at TG curve (Figure-3) of Cu(II) complexes of show that, the complexes stable up to 160 °C. Thereafter, the complexes start decompressing gradually. The percentage weight loss is about 3.40% in complex (cal 3.5%). This decomposition begins at 160 °C and continues up to 240 °C. The weight corresponds to two mole of coordinated water molecule. The loss of coordinated water molecules in Cu(II) complex is a single step process, indicated by endothermic peak in DTA curve. After this, Cu(II) complexes show sharp decrease in weight, indicating decomposition of complexes, further confirmed by endothermic nature of peaks obtained in DTA curve, organic part content of meal complex is removed during this loss and decomposition continues up to the temperature 800 °C leading to the formation of stable residue of metal oxide i.e. CuO.

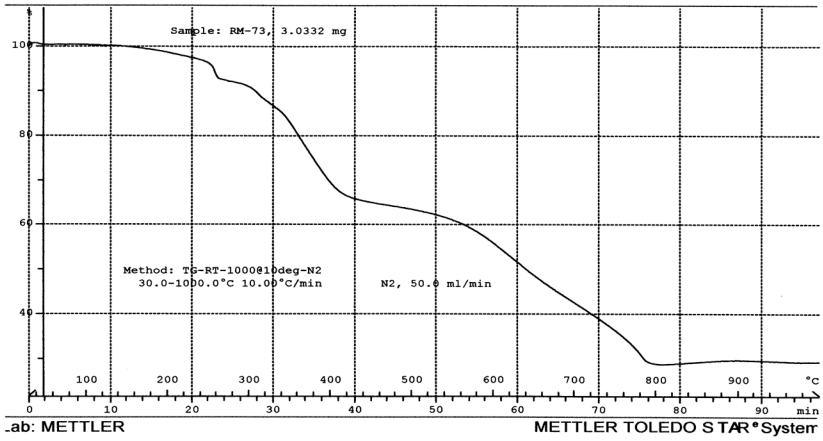


Figure:3 (a)TG Curve of Cu complex

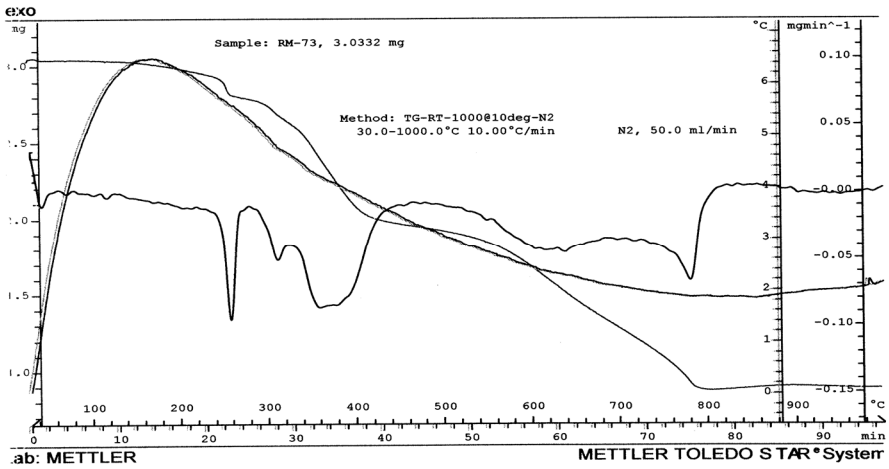


Figure:3 (b)TG-DTA DTG Curve of Cu complex

The simultaneous use of TG and DTA show that the metal complexes are thermally quite stable. Thermograms of all the complexes indicate the presence of coordinated water molecules. X-Ray powder pattern investigation suggests that all the complexes are crystalline in nature.

A. Antimicrobial Activity

The ligand and their metal complexes were tested for antibacterial activity Mueller Hinton agar was used for testing the susceptibility of microorganism by well diffusion method, using DMSO as solvent, at a concentration of 0.01 M against gram positive (staphylococcus aureus) and gram negative (Escherichia coli) bacteria.

The zones inhibition against the growth of microorganisms was determined at the end of incubation period 24 h at 37^{o}C and the results are presented in table 3. It was found that the metal complexes are more active than the free ligand.

III. ACKNOWLEDGMENT

The author is thankful to Dr. W N. Jadhav, Principal Sharda Mahavidyalaya Parbhani, Dr. B. C. Khade, Dr. S.R. Bhusare, DSM College, Parbhani, Dr. B. R. Patil, Sharda Mahavidyalaya , Parbhani for their valuable guidance. Also thanks to Dr S. L. Sadawrte, Principal, DSM College, Jintur, for providing the necessary facilities.

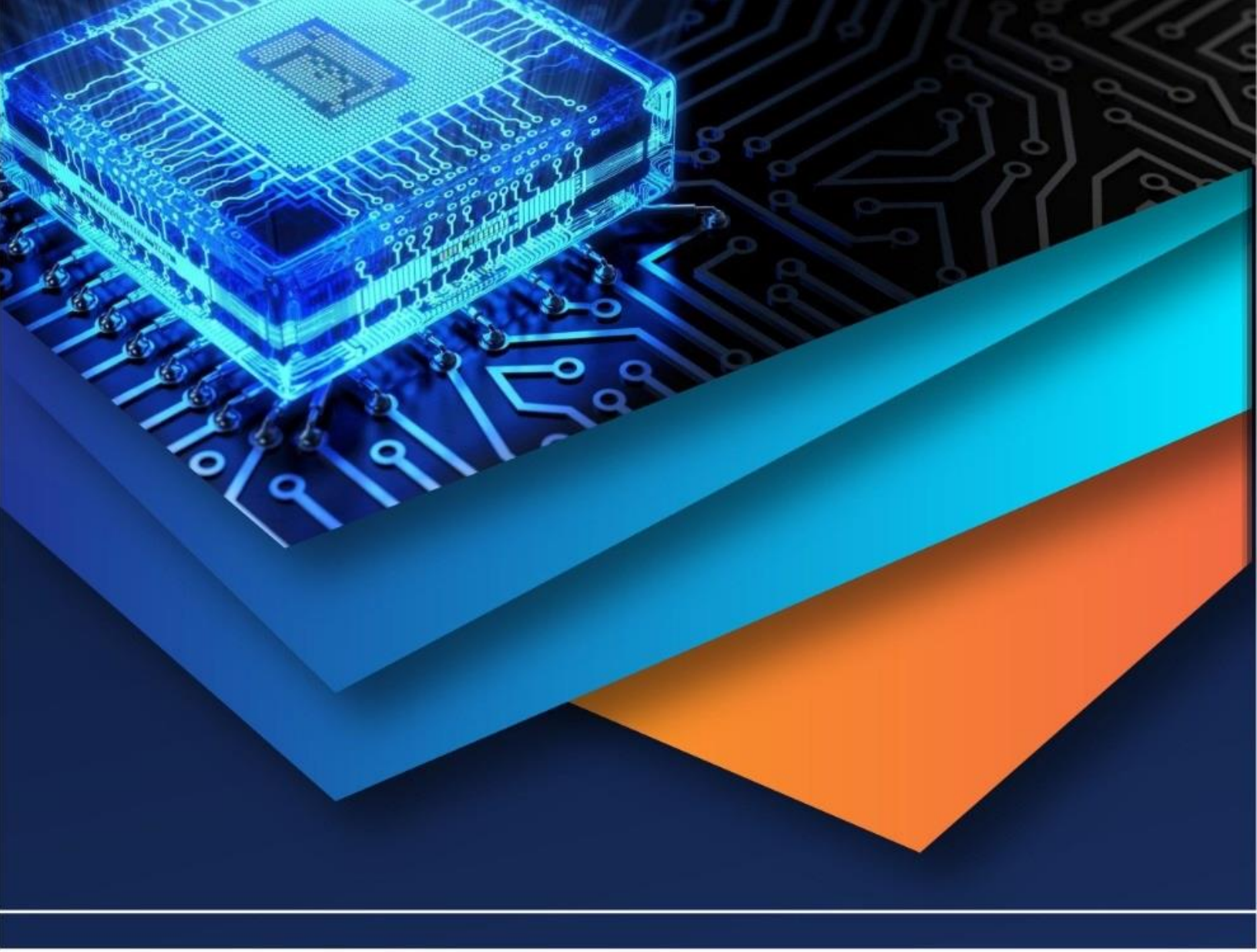
Table 3: Antimicrobial activities of Schiff bases and their complexes

compound	Zone of inhibition	
	Staphylococcus aureus	Escherichia coli
L	12	11
L-Cu	16	17
L-Ni	15	14
L-Co	17	18
L-Zn	14	13
Standard drug	**26**	**30**

REFERENCES

[1] Antloin L., Febretel A C, Galtesti D, Giushi A and Sessoli R, Inorg.Chem,29, 143(1990) .
[2] Jons,R.D.,Summerville, D.A.and Basolo,F.,Chem.Rev,79,130(1979).
[3] Sudha Goyal and Keemati Lal. J. Indian Chem. Soc., 66-477 (1989).
[4] Wu. Zishen, Gui Ziqui and Yen Zhenhuan, Synth. React. Inorg. Met org. Che.20.335 (1990)
[5] R.K. Parashar, R.C. Sharma, Anil Kumar and Govind Mohan, Inorg. Chim. Acta.151,201 (1988)
[6] Merchant, Jaysukhal R., Chothia D.S., J. Med. Chem., 13 (2), 335-36 (1970)
[7] M. Kumar, Orien. J. Chem.18(3) 559(2002)
[8] Geary, W.J., Coord. Chem, Rev, 7, 82(1971).
[9] Silverstein, M. R., Bassler, G. C. and Morril, T. C., Spectrometric Identification Of Organic Compounds, John Wiley and Sons,4th ed., 1981,p-111,130.
[10] Rao, N.R., Venkateshwar, P.R., Reddy. G.V. and Ganorkar, M.C., Indian J. Chem., 26A (10), 887 (1987).
[11] Mohamad, G. G., Omar, M. M. and Hindy, A. M. Turk. J. Chem., 30,361,2006.
[12] Nakamoto, K., "Ingrared Spectra of Inorganic coordination compounds", John Wiley Publication, New York (1966; 1970)
[13] Dash, D.C., Pansa, A.K., Jena, P., Potioshi, S.B. and Mahapatra A., J. Ind. Chem. Soc., 79, 48-50 (2002).
[14] Sutton, D., "Electronic Spectra of transition metal complexes" McGrow Hill, 145-148 (1968
[15] Mahapatra, K., Rupini, B and Srihari, S., J. Ind. Chem. Soci, 81, (2004) 950-953
[16] Feggis, B.N. and J. Lewis, Prog. Inorg. Chem., 6, 87 (1964).
[17] Dinkar P Kotwal.,W. N. Jadhav., "Synthesis, Characterization and Antimicrobial study of some Cu(II), Ni(II)Co(II) and Zn(II) Bidentate Schiff base metal complex" Vol. 3,Issue 2,91-94,IJUS,ISSN-2454-7263(Jan 2018)
[18] Freeman and Carroll, J. Phy. Chem. 62, 391, 1958
[19] Aswar, A. S. J. Indian Chem. Soc. 75, 395, 1998.
[20] Dinkar P. Kotwal, IJESRT. ISSN: 2277-9655 ,10(5), Impact Factor: 5.164, IC™ Value: 3.00 CODEN: IJESS7, Page 57 61, May, 2021

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



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)