ORIGINAL RESEARCH PAPER
Volume - 13 | Issue - 12 | December - 2024 | PRINT ISSN No. 2277 - 8179 | DOI : 10.36106/ijsr
INTERNATIONAL JOURNAL OF SCIENTIFIC RESEARCH

SYNTHESIS, CHARACTERIZATION, AND INVESTIGATION OF SCHIFF BASES IN RELATION TO THEIR ANALYTICAL USE

Chemistry

Prof. Ms. Pratiksha G. Wagh Msc. In Analytical Chemistry

KEYWORDS

INTRODUCTION

A Schiff base, named after Hugo Schiff, is a compound with a functional group that contains a carbon-nitrogen double bond with the nitrogen atom connected to an aryl or alkyl group, not hydrogen. Schiff bases in a broad sense have the general formula R1R2C=NR3, where R is an organic side chain. In this definition, Schiff base is synonymous with azomethines. Some restrict the term to the secondary aldimines (azomethines where the carbon is connected to a hydrogen atom), thus with the general formula RCH=NR'. The chain on the nitrogen makes the Schiff base stable imines. A Schiff base derived from aniline, where R3 is a phenyl or a substituted phenyl. Schiff bases are formed when any primary amine reacts with an aldehyde or a ketone under specific conditions. Structurally, a Schiff base (also known as an imines or azomethines) isa nitrogen analog of an aldehyde or ketone in which the carbonyl group (C=O) has been replaced byan imines or azomethines group. Schiff bases are some of the most widely used organic compounds. We also highlight the most significant examples of compounds belonging to this class, which exhibit analgesic, anti-inflammatory, antimicrobial and non- ulcerogenic activities to have been reported in the literature. Schiff bases are crystalline or oily substances that are insoluble in water and soluble inorganic solvents. They are weak bases, forming salts with acids in an anhydrous medium; in aqueous acid solutions, they undergo hydrolysis to yield an amine and aldehyde. The majority of Schiff bases are stable in alkaline solutions. Schiff bases are valuable intermediate products of organic synthesis, for example, in the preparation of secondary amines and various heterocyclic compounds. The Schiff bases known as azomethines dyes are used for dyeing acetate and synthetic fibers; they are also used in color photography to reduce the photosensitivity of photographic emulsions.

These have several applications in organic studies, such as for building new heterocyclic systems, for identification, detection, and determination of aldehydes and ketones, for purification of carbonyl or amino compounds, or for the protection of these groups during the complex formation or such sensitive reactions. They have other side applications in various other fields, coordination chemistry, analytical chemistry, pigments and dyes, and polymer industries, in vitamins and enzymes for model biomolecules. There is a special mention of these complexes in agriculture as fungicides, pesticides, and bacteriocides. Survey of the literature for SB metal complexes and their applications showed excellent review articles for the detailed understanding of this class of compounds in all respects and one more especially dedicated to copper complexes. They provide several details on number of metal complexes derived from SBs used widely for applications in food and dye industry, analytical chemistry, catalysis, polymers, antifertility, agrochemical, anti-inflammatory activity, antiradical activities, and biological systems as enzymatic agents. Several have reviewed them in light of their antimicrobial, antibacterial, antifungal, antitumor, and cytotoxic activities. There are some individual articles too not mentioned in them with studies on the above mentioned types of activities with some metals ions such as Cu(II), Ni(II), and Co(II) with SB derived from salicylaldehyde and 2-substituted aniline. Cobalt(II) complexes with thiosemicarbazone found potent antitumor agents. Mn(II), Ni(II), Cd (II), Zn(II), Cu(II) Complexes of 3-(4-hydroxyphenyl)-1-phenyl-1h-pyarazole-4- carbaldehyde 4N-(2-pyridyl) thiosemicarbazone Showed good cytotoxic activity while cobalt complex showed better antioxidant activity.

Schiff base metal complexes have been studied extensively because of their attractive chemical and physical properties and their wide range of applications in numerous scientific areas. These types of complexes have been vigorously explored in recent years and such studies have been the subject of many papers and reviews.

MATERIALAND METHOD: -

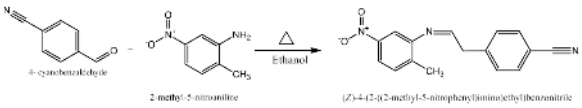
All the chemicals of Sigma Aldrich were purchased, and solvents were distilled before use. Melting point was recorded on DBK digital melting point apparatus. ^{1}H NMR, IR and Mass spectra were recorded at SAIF, Punjab University, Chandigarh, India.

Synthesis Of Ligand:-

1) (E)-4-bromo-N-(5-Bromo-2-methoxybenzylidene)- 2-chloroaniline:

(E)-4-bromo-N-(5-Bromo-2-methoxybenzylidene)--2-chloroaniline was synthesized asper known procedure (Gupta et al).4-Bromo-2-Choroaniline (0.51gm) dissolved in 8 to 10 ml ethanol in hot condition. Then 0.53 gm5-Bromo-o-anisaldehyde dissolved in ethanolwas added and kept reaction mixture for refluxing with stirring for 8 to 9hrs. The reaction was monitored by TLC. The reaction mixture was poured on ice cold water with constant stirring (20 to 25 min.) by which crystals of the product was formed. Then filtered and dried. The ligand is soluble in ethanol.

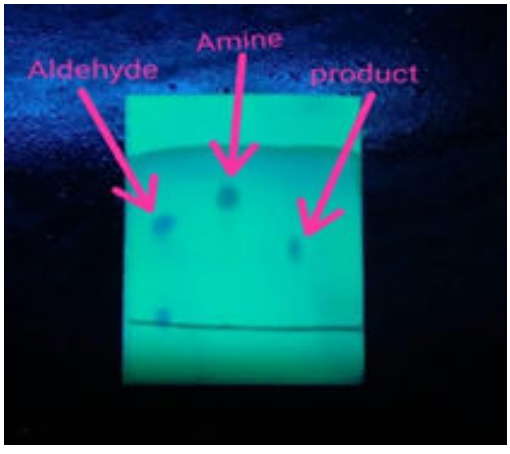
Reaction Scheme:



RESULT AND DISCUSSION:

(E)-4-bromo-N-(5-Bromo-2-methoxybenzylidene)-2-chloroaniline the reactions were monitored by thin layer chromatography and the products isolated were single spot .

Compound Table – represent the Molecular weight, molecular formula, Melting point & % yield of all the synthesized compound.



Thin Layer Chromatographs:

Thin Layer Chromatographs:

(E)-4-bromo-N-(5-Bromo-2-methoxybenzylidene)- 2-chloroaniline

Stationary Phase: silica
Mobile Phase: n-hexane and ethyl acetate (8:2ratio)

FTIR Spectra For Ligands

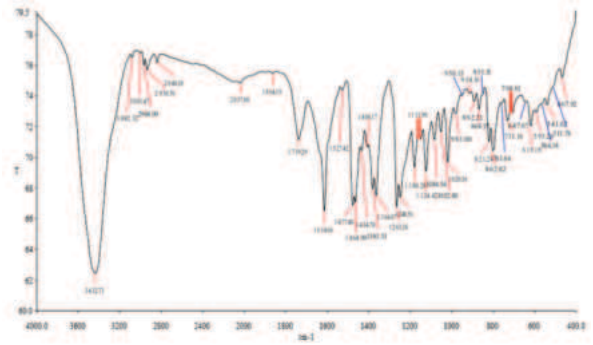


Fig.-

FTIR Spectrum of (E)-4-bromo-N-(5-Bromo-2-methoxybenzylidene)-2-chloroaniline

All the synthesized compounds were confirmed by FTIR spectra. FTIR data of (E)-4-bromo-N- (5-Bromo-2-methoxybenzylidene)- 2-chloroaniline compound is Following:

- ν (OH) in cm^{-1} = 3245
- ν (C=N) in cm^{-1} = 1603
- ν (R-C-N) in cm^{-1} = 1168
- ν(Ar-O) in cm^{-1} = 1368

Mass Spectroscopy:-

All compounds were studied with mass spectroscopy and found confirmed formation of compounds. The peaks obtained showed good agreement between theoretical and observed values.

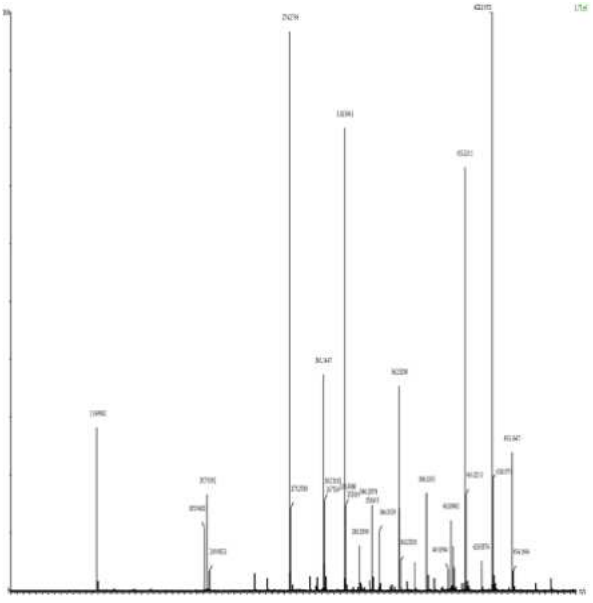


Fig.-

Mass spectrum of(E)-4-bromo-N-(5-Bromo-2-methoxybenzylidene)-2-chloroaniline

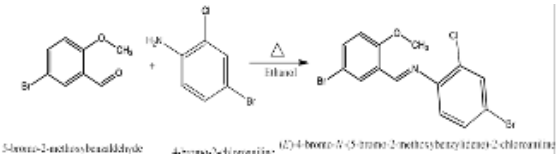
The molecular weight have been confirmed with mass spectrum

Observe Data -
Calculated Mass:403
Observed Mass: 402

2) (Z)-4-((2-methyl-5-nitrophenyl) amino) methyl) benzonitrile:

(Z)-4-((2 -methyl-5-nitrophenyl) amino) methyl) benzonitrile was synthesized as perknown procedure 2-methyl-5-nitroaniline (0.10gm) dissolved in 8 to 10 ml ethanol in hot condition(Guptaetal).Then 0.65 gm 4-Cyanobenzaldehyde dissolved in ethanol was added and kept reaction mixtureforrefluxing with stirring for 8 to 9 hrs. The reaction was monitored by TLC. The reaction mixturewas pouredon ice cold water with constant stirring (20 to 25 min.) by which crystals of the productwas formed.Then filtered and dried. The ligand is soluble in ethanol.

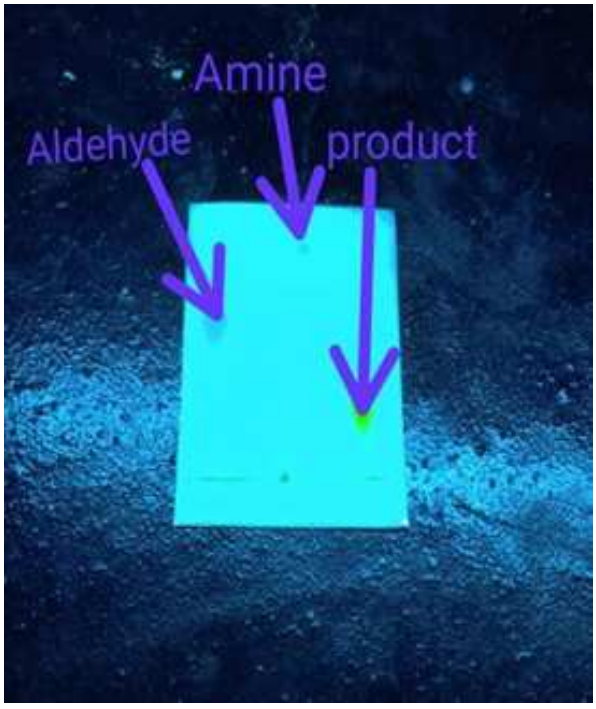
Reaction Scheme :



RESULT AND DISCUSSION-

(E)-N-(5-bromo-2-methoxybenzylidene)-2-methyl- 5-nitroanilinereactions were monitored by thin layer chromatography and the products isolated were single spot .

Compound Table – Represent the Molecular weight, molecular formula, Melting point& % yield of all the synthesized compound.



Thin Layer Chromatographs:

(E)-N-(5-bromo-2-methoxybenzylidene)-2-methyl- 5-nitroaniline
Stationary phase: silica
Mobile phase: n-hexane and ethyl acetate(8:2ratio)

FTIR Spectra For Ligands

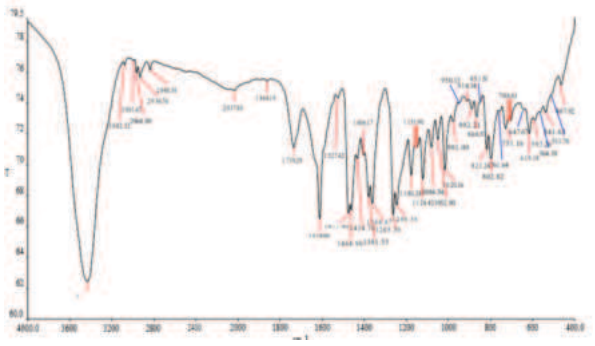


Fig-

FTIR Spectrum of (E)-N-(5-bromo-2-methoxybenzylidene)-2-methyl- 5nitroaniline

All the synthesized compounds were confirmed by FTIR spectra. FTIR data of (E)-N- (5-bromo-2-methoxybenzylidene)-2-methyl-5nitroanilinecompound isFollowing:

- ν (OH) in cm-1 = 3410
- ν (C=N) in cm-1= 1612
- ν (R-C-N) in cm-1= 1174
- ν (Ar-O) in cm-1 = 1376

Mass Spectroscopy:-

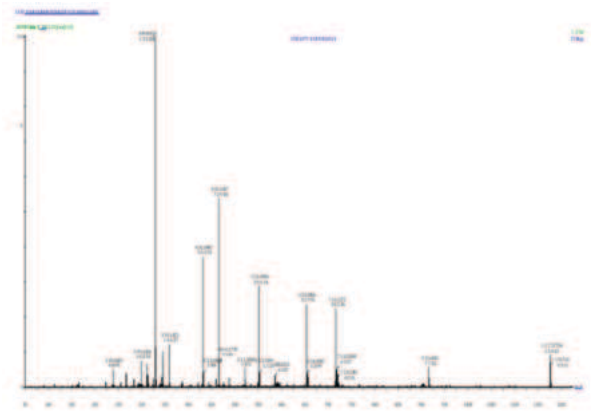


Fig-
Massspectrumof(E)-N-(5-bromo-2-methoxybenzylidene)-2-methyl-5-nitroaniline:
The molecular weight have been confirmed with mass spectrum.

Observe data.-
Calculated mass:349
Observed mass:349

Data of Synthesized Compounds:

Sr. No.	Name of the Compound	Molecular Formula	Molecular Weight	M.P.	Nature and Colour	%Practical Yield
1	(E)-4-bromo-N-(5-bromo-2-methoxybenzylidane) 2-aniline	C14H10Br 2ClNo	402	231° C	Yellow	60.00
2	(Z)-4-((2-methyl-5-nitrophenylamino)methylbenzonitrile.	C18H13N 3 O2	277	216° C	White	52.38
3	(E)-N-(5-bromo- 2-Methoxybenzylidene)- 2-methyl-5-nitroaniline	C15H13Br N2O3	349	248° C	slightl	55.00

CONCLUSIONS:
- Three Ligands have been synthesized.
- All synthesized compounds have been characterizedby, FTIR and Massspectroscopic techniques.
- Significant antimicrobial activity, suggesting applications in pharmaceuticals.
- Distinct optical properties, useful for optical devices and materials.
- Further extension work regarding anti micro bial studies will be carried out in future.

Acknowledgement
It gives me a great pleasure to express my deepest sense of gratitude towards my guide Dr. A. M. Patil for his inspiring explanation and most competent guidance for successful completion of this project.It was a golden opportunity for me to work under his guidance. He will be remembered forever for this great job.

I would like to express my thankful regards to 'Jain Gurukul' and our honorable Principal Dr. G. H. Jain for providing necessary facilities for carrying out this work. I am especially thankful to our Head of Department Dr. R. S. Sancheti for his valuable discussions at the time ofexperimental work.

I wish to express my thanks toMs. Sajeda Sayyad, Dr. V. S. Aware, Mr. S. P. Khairnar, Dr. H. H. Shah and all teaching staff for their co-operation during the completion of this project.

I wish to express my thanks to our nonteaching staff members Mr. D. S. Shinde, Mr. S. B. Chaudhari, Mr. K. S. Nikam, & Mr. T. S. Jadhav for their kind support and extended co-operation in making my project successful.

I also express my special gratitude to my dear colleagues Ms. Pranjal pawar, Ms. Meghna Kahadne, Mr. Tushar Ghodke who worked with me for some months & my beloved parents who inspired, nurtured and rewarded adream to excel in Chemistry, without their love and sacrifice dream would not have been a reality.

Last but not the least, we are thankful to each and everyone who directly or indirectly supported us and encouraged us to complete this work smoothly & successfully.

REFERENCES
[1] Nic M, Jirat J, Kosata B eds (2006) Schiff base IUPAC Compendium of Chemical Terminology. DOI:10.1351/ goldbook. S05498. ISBN 096785 9-8.
[2] Przemysławiyal S., Rehman W., BalochM. K., Muhammad B., Badshah A. Khan K. M.: 18. Characteristic spectral studies and invitro antifungal activity ofsome Schiff bases and their organotin (IV) complexes. Chin. Sci. Bull., 2004, 49, 2, 119–122
[3] Nic M, Jirat J, Kosata B eds (2006) anils. IUPAC Compendium of Chemical Terminology (DOI:10.1351/goldbook.A00357. ISBN 0-9678550-9-8.
[4] Schiff H (1846)Arm Chem137:118.
[5] Da Silva CM, Da Silva DL, Modolo LV, RosemeireBAlves, MariaA de Resende, et al.(2011) Schiff bases: A short review of their antimicrobial activities.J of adv res2(1): 1-8.
[6] S. Patai, "The chemistry of carbon-nitrogen double bond," in General and Theoretical Aspects, C. Sandorfy, Ed., chapter 1, pp. 1–60, Inter science, William Clowes and Sons, London, UK, 1970.
[7] F. P. Dwyer and D. P. Mellor, Chelating Agents and Metal Chelates, Academic Press, New York, NY, USA, 1964.
[8] H.Holm, G.W. Everett ,and A. Chakraborty, "Metal complexes of Schiff bases and ketoamines," Progress in Inorganic Chemistry, vol. 7, pp. 83–214, 1966.
[9] J. W. Smith, The Chemistry of Carbon-Nitrogen Double Bond, edited by S. Patai, International Science Publishing, London, UK, 1970.
[10] L. F. Lindoy, "Metal-ioncontrolinthe synthesis of Schiff base complexes," Quarterly Reviews, Chemical Society, vol. 25, no. 3, pp. 379–391, 1971.
[11] B. O. West, E. A. V. Ebsworth, A. G. Maddock, and A. G. Sharp, New Pathways in Inorganic Chemistry, Cambridge University Press, London, UK, 1966. 20 International Journal of Inorganic Chemistry
[12] S.Yamada,"Recent aspects of the stereochemistry of Schiff base-metal complexes," Coordination Chemistry Reviews, vol. 1, 4,pp. 415–437, 1966.
[13] M. J. Norgett, J. H. M. Thornley, and L. M. Venanzi, "The visible and ultraviolet spectra of d6 -, d7 - and d8 -metal ions in trigonal bipyramidal complexes," Coordination Chemistry Reviews, vol. 2, no. 1, pp. 83–98, 1967.
[14] N. F. Curtis, "Macrocyclic coordination compounds formed by condensation of metal-amine complexes with aliphatic carbonyl compounds," Coordination Chemistry Reviews, vol. 3, no. 1, pp. 3–47, 1968
[15] E. Bayer, "Structure and specificity of organic chelating agents," AngewandteChemie International Edition, vol. 3, no. 5, pp. 325– 332, 1964.
[16] E. Jungreis and S. Thabet, "Analytical application of schiff bases," in Chelates in Analytical Chemistry, H. A. Flaschka and A. J. Barnard Jr., Eds., vol. 2, pp. 149–177, Marcel Dekker, New York, NY, USA, 1969.
[17] F. Feigl, Spot Tests inorganic Analysis ,vol.2,Elsevier, NewYork, NY,USA,1958.
[18] J. C. Bailar, Chemistry of Coordination Compounds, Reinhold,1956.
[18] S. Chaberek and A. Martell, Sequestering Agents, John Wiley & Sons, New York, NY, USA, 1959.
[19] R.L. Smith, The Sequesteration of Metals: Theoretical Considerations and Practical Applications, Chapman & Hall, London, UK, 1959.
[22] E.R. Inmanand I.A. Macpherson, Chemical Abstracts, vol.74,ArticleID77398, 1971.
[20] C. S. Marvell and N.Tarkoy,"Heat stability studies on chelates from schiff bases of salicylaldehyde derivatives. II," Journal of the American Chemical Society,vol.80, no. 4,pp.832–835,1958.
[21] W. Qin, S. Long, M. Panunzio, and B. Stefano, "Schiff bases: a short survey on an evergreen chemistry tool," Molecules, vol. 18, no. 10, pp. 12264–12289, 2013.
[22] S. Kumar, D. N. Dhar, and P. N. Saxena, "Applications of metal complexes of Schiff bases—a review," Journal of Scientific and Industrial Research, vol. 68, no. 3, pp. 181–187, 2009.
[23] R. Katwal, H. Kaura, and B. K. Kapur, "Applications of copper— Schiff 's base complexes :areview," Scientific Reviews & Chemical Communications, vol. 3, no. 1,pp.1– 15, 2013.
[24] R. K.Parashar, R. C. Sharma, and G.Mohan, "Biological activity of some Schiff bases and their metal complexes," Biological Trace Element Research, vol. 23, pp. 145–150, 1990.
[25] Schiff, Ugo (1866). "Sopra una nova seriedibasiorganiche" [On a new series of organic bases]. Giornale di Scienze Naturali ed Economiche (in Italian). 2: 201–257.