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Synthesis and Characterization and Biological Activities of Hydrazones

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ABSTRACT: Hydrazones derivatives of carbonyl compounds constitute an important class of biologically active compounds. Metal complexes of Schiff bases represent an important class of coordination compounds. Schiffs base containing hetero donar atom continue to provide the interesting facts in the fields of coordination chemistry.

Literature studies on hydrazones have shown that these derivatives possess a wide variety of biological activities such as anti-tumour, anti-bacterial, anti-viral, anti-hypertensive, analgesic etc. Complexes of Co(II), Ni(II) and Zu(II) with schiff base [LH_2] derived from 2- hydroy – 5 – Bromo -3- Nitro acetophenone and isonictonoyl hydrazide have been synthesized and characterized on the basis of molar conductance magnetic properties, elemental analysis, infrared, ^{1}H NMR, electronic spectra and thermo gravimetric analysis. The schiff base acts as mono basic bi-dentate ligand commonly co-ordinates through the oxygen atom of the deprotonated phenolic group and the nitrogen atom of azomethine group, which is confirmed by IR spectral data. Thermal analysis indicates the coordinated and lattice water molecules in the complexes which are also I.R. spectral data.

KEYWORDS: Schifft bare, Magnetic Susceptibility, Biological activates.

I. INTRODUCTION

Schiff bases are an important class of ligand in coordination chemistry. Schiff bases derived from hydrazones and aroyl hydrazines have been widely used as ligand for the synthesis of transition metal complexes. Schiff bases or their metal complexes have many applications in different fields Hydrazones, heteroaroyl hydrazones ligands and their metal complexes are biologically active. Heteroaroyl hydrazones forms stable metal complexes with transition metal ions and inner transition metal ions due to complexing ability of ligand through keto-enol tautomerism and availability of other donar sites in the ligand i.e. isonicotinoyl hydrazide is one of the drug [n chemotherapy of tuberculosis. Due to its biological potency, pharmacological properties and synthetic flexibility of Schiff base derived from isonicotinic acid hydrazide. The aim of present investigation is to synthesize various transition metal complexe of Schiff base derived from 2-hydroxy-5-Bromo -3-nitroacetophenone and isonicotinoyl hydrazide.

II. EXPERIMENTAL

All the chemicals used were of AR grade and used as received isonicotinoyl hydrazide (IH) was obtained from E.Merck. 2-hydroxy-5-chloro- 3-nitro acetophenone (HCNA) was prepared by known method. The solvents were purified by standard methods.

Synthesis of 2-hydroxy-5-Bromo-3-nitroacetophenone isonicotinoyl hydrazone [HCNAIH]

A solution of isonicotinoyl hydrazide (0.01M) in' 12.5% of ethanol was added to an ethanolic solution (12.5) of 2-hydroxy-5-Bromo-3-nitroacetophenone (0.01M) and the reaction mixture was refluxed on a water bath for 2 ½. Then cooled to 25^{0}C. The resulting pale yellow coloured solid was washed with ethanol, crystallized from DMF and dried under reduced pressure at ambient temperature. The purity of ligand was checked by elemental analysis and melting point. It was also characterized by IR and ^{1}H NMR spectral studies. Yield: 75% MP. 268-270 ^{O}C.

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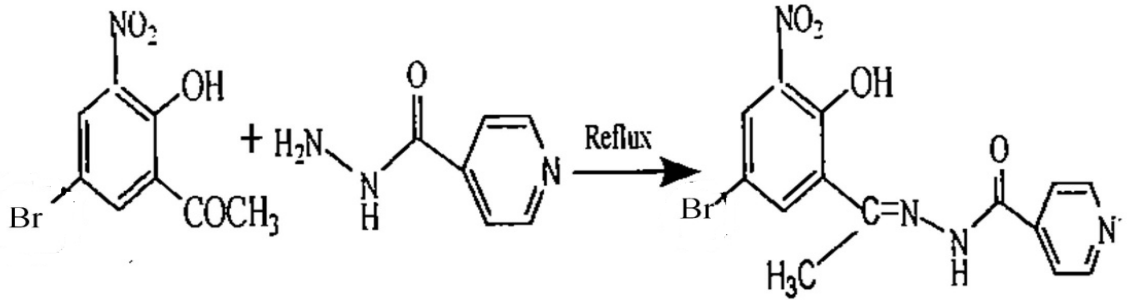

isonicotinoyl hydrazide
2-hydroxy-5-Bromo -3-nitroacetophenone 2-hydroxy-5-Bromo-3-nitroacetophenone isonicotinoyl hydrazide

Scheme I-Synthesis of the ligand (LH_3)

Preparation of complexes

All the metal complexes were prepared in a similar way by following method. To a hot solution of ligand HCNAIH (0.01M) in 12.5ml of ethanol a suspension of respective metal salts [acetates of Co (II), Ni (11), Cu (11), and Zn (II)] was added drop wise with constant stirring. The reaction mixture was refluxed on a water bath for 4-6h. The precipitated complexes were fi1tered washed with ethanol. Followed by ether and dried over fused calcium chloride. Yield: 45-50%.

The complexes are soluble in DMSO and DMF but insoluble in water and common organic solvents. The metal chloride content of complexes was analyzed by standard methods. The HNMR spectra of ligand was recorded and obtained from RSIC Chandigarh. IR spectra of the: compounds were recorded on Perkin Elmer 842 spectrophotometer in the region 400-4000cm Carbon, Hydrogen and Nitrogen analysis were carried out at RSIC Punjab University, Chandigarh. The molar conductance of the complexes at 10^{-3}M dilution in DMF were determined using equiptronic digital conductivity meter EQ-660. with a cell constant 1.00 cm^{-1} at room temperature. The magnetic moment measurement were made on a - Gouy balance at room temperature using [HgCo $(SCN)_4$] as the calibrant. The thermo gravimetric analyses were performed on laboratory set up apparatus in air atmosphere at 10^0 mm^{-1} heating rate. The molecular weights of the complexes were determined by Rast method.

III. RESULTS AND DISCUSSION

The ligand HCNAIH and its complexes have been characterized on the basis of ^{1}H NMR, IR spectral data, elemental analysis, diffused reflectance spectra, molar conductance, magnetic
succeptibility, measurements and thermogravimetric analysis data.

The ^{1}H NMR spectra of ligand HCNAIH shows signals at 8 12.95, (1H, s, phenolic OH), 11.34, (1H, s, imino); 9.05 and 9.08 (4H, d, isonicotine); 8.15 and 7.35, (2H, m, phenyl) and 3.25 ppm, (3H, s, methyl). All these values and analytical data are consistent with proposer molecular formula of ligand. All the compounds; are coloured solid and stable in air. All the compounds are insoluble in water but soluble in coordinating solvents like DMF and DMSO. The molar conductance values in DMF (10^{-3} M) solution at room temperature (Table.2) shows all the complexes are non electrolytes. IR spectra of ligand shows V(C=N) peaks at 1617 cm^{-1} and absence of C=O peak at around 1700 — 1750 $cm^{-1,}$ indicates the Schiff base formation. Other reported peaks are V(N-H) at 3182 cm^{-1}, V(OH) at 2991 cm^{-1} V(C=O) phenolic at 1531 cm^{-1}, pyridyl ring breathing peak at 1070 cm-1, V(N-N) peak at 991 cm^{-1} and V(C=N) peak at 1617 cm^{-1}, The V(C=N) peak of ligand is found to be shifted to lower frequencies by 37-47 cm^{-1} in the spectra of complexes indicating the coordination via the azomethine nitrogen, which is also confirmed by appearance of bands in the range of 550 — 345 cm-’ which have been assigned to V(M-N) band.

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Thermogravimetnic studies

Thermogravimetnic study indicates all the complexes are stable up to 60-70°C. All the complexes except Cu (II) show two stage decomposition patterns and Cu (II) show three stage decomposition pattern. The percentage weight loss data (Table 3) up to 140°C indicates the loss of one water molecule from Co (II) and Ni (II) complexes each, loss of two water molecule from Zn (II) complexes and loss of three water molecules (lattice) from Cu (II) complexes each. Further loss in weight up to 220-240°C was observed. The percentage weight loss data indicates loss of one coordinated water molecule from Ni (II), complexes each, loss of two coordinated water molecule from Co (II) and Zn (II) complexes each. There is no weight loss at 220-240°C in Cu (I!) complexes indicates the absence of coordinated water molecule in this complex.

Antimicrobial activity

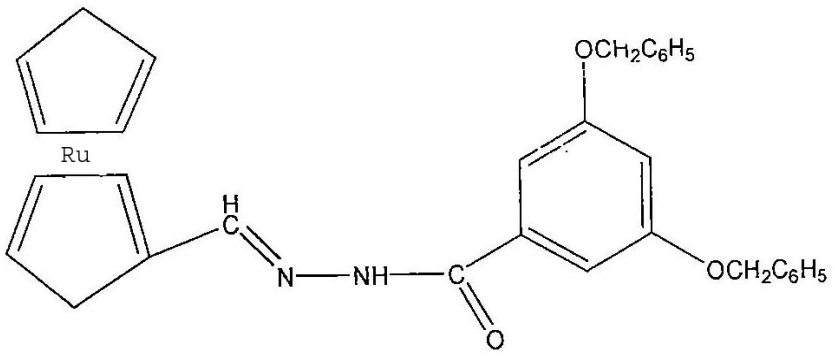
The compounds are found to show low bactericidal behavior against most of the bacterial culture and resistance towards the other. In general the results reveal that the activity of the ligand was found to enhance on complexation with metal. The inhibition effect of the ligand and its metal complexes on the growth of various bacteria is summarized. In an urge to develop new antimicrobial compound, a number of hydrazones were tested for their antimicrobial activities because of the evolution of drug-resistant microbial pathogens.

Some derivatives of flavanol hydrazones were synthesized and screened for their in vitro anti-bacterial activity against 25 strains of Gram –ve and Gram +ve pathogenic bacteria. The synthesized compound demonstrates inhibitory effect (MIC< 392 μ g/ml) against few pathogenic bacterial strains. The hydrazones possessed activity against methicillin-resistant Staphylococcus aureus strain may be due to the presence of carbonyl region and hydroxyl group.

A series of quinoxaline derivatives was synthesized and evaluated for their antimicrobial activity. The compounds which were bearing highly electronegative chloro and fluoro substituents at the para position of phenyl ring exhibited good activity as compared to those compounds having these atoms at either ortho or meta position or the other compounds containing the less electronegative! electropositive substituent at these positions.

Thirty new hydrazones of 1-phenyl, 1-benzyl and 1-benzhydryl - 4 - amino piperazines were tested for antibacterial activities against E.coli, Staphylococcus aureas, B. subtillis an anti fungal activities against Candida albicans and Saccharomyces cerevisiae. Among thirty hydrazones, 1-benzhydryl- 4 -isonicotinylidene amino piperazine showed a broad spectrum of activity.

A new chelating ligand. (1-formyl Ruthenocene)-3,5-dibenzyloxybenzoyl hydrazone (HL) la and three transition metal complexes, ML2 [M 5 Cu(II), Ni(II), Zn(II) were synthesized by Lin et.al. They evaluated antibacterial activities of compounds. Preliminary studies indicated that the ligand and its three complexes were active against S.aureus, but were uneffective against E.coil



Some N- (1-benzyl-2-phenylethylidene)-N'-[4-(aryl] thiazol-2-yl hydrazone and N-(1-penylbutylidene)-N' -[4-(aryl) thiazol-2-yl] hydrazones derivatives were synthesized and evaluated for antifungal activity. Their antifungal activities against standard and clinical strands of Candida albcans, Candida alabrata, Candida utilis, Candida tropicalis, Candida kruest, Candida zeylanoides, and Candida parapsilosis were observed and were found to be significant.

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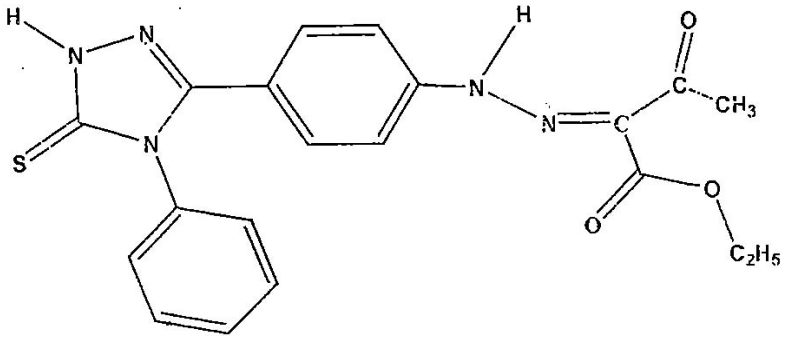
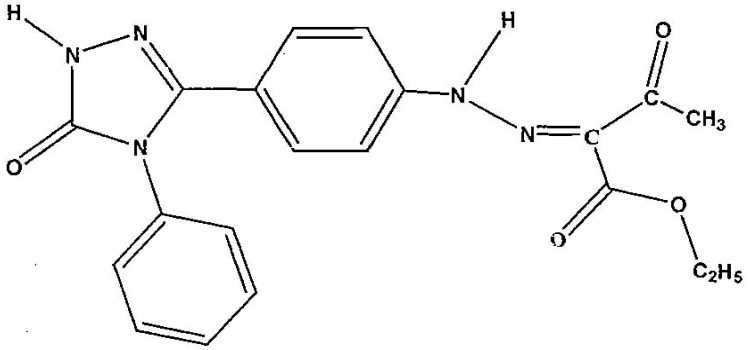
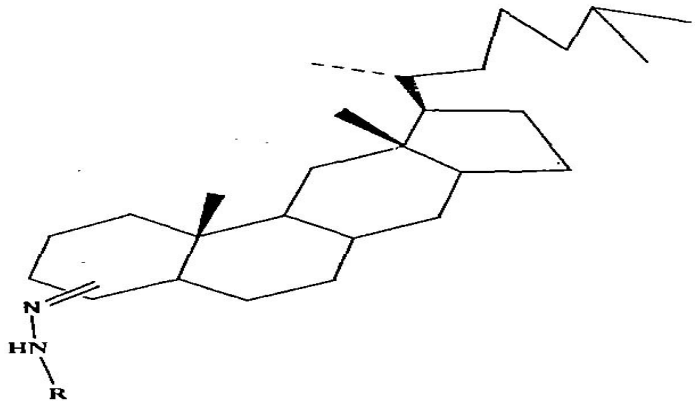
Ethyl 2-arylhydrazono-3-oxobutyrates were synthesized in order to determine their antimicrobial properties. Compound 2a showed good activity against S, aureus whereas the others had no remarkable activity on this strain. Compound 2b was found to be more active than the other compounds against mycobacterium fortuitum at a MIC value o 32 μ g /mL.





A series of hydrazones 3 synthesized from various cholesterol derivatives were screened for their in vitro antimicrobial properties against human pathogens.



Antihypertensive activity

M. Minami et.al. elucidated the effects of a new vasodilating antihypertensive drug budralazine 4, mesityl oxide-1-phthalazinyl hydrazone on drinking behavior of water and humoral factors including plasma norepinephrine(NE), angiotension II (A II), arginine vasopression (AVP), serotonim (5-HT) concentrations, urinary aldosterone and

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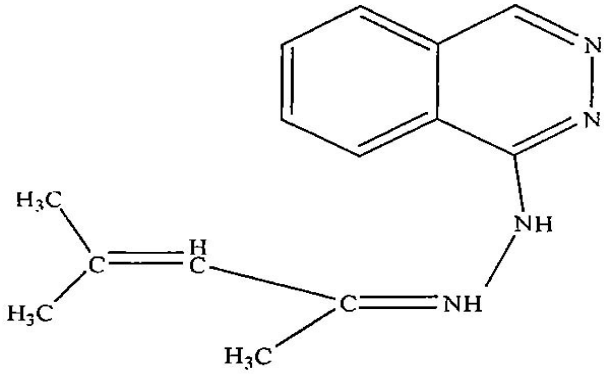
catecholamine excretion rates in rats. The results suggested that budralazine it active on rennin angiotensin aldosterone system in comparison to sympathetic nervous system



Budralazine
Mesityl oxide-1-phthalaziny hydrazone

Anticonvulsant activity

Epilepsy is most common neurological disorder, second to, stroke. The number of drugs useful for the treatment of epilepsy is remarkably small. New epileptic drugs have been developed that may constitute novel and effective t therapies or epilepsies.

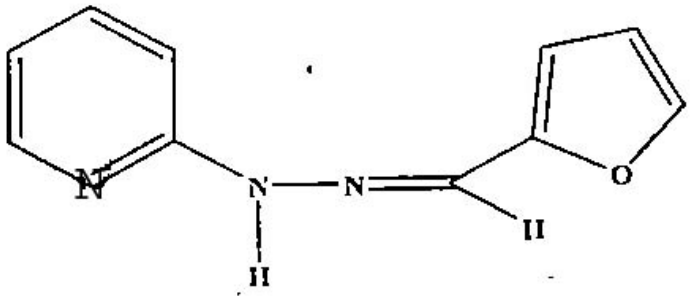
It was found that both 2- oxobenzoxazoline and 2- oxobenzothiazoline hydrazone derivatives exhibited remarkable anticonvulsant activity. 5- chloro – 2 –(3H) – benzoxazoline – acetyl – 2 – (o-methybenzaldehyde) hydrazone, 5- chloro – 2 –(3H) – benzoxazolinone -3- acetyl – 2 – (pmethylbenzaldehyde) – 3 – acetyl – 2 – (p – nitrobenzaldehyde) – hydrazone, and 5 – chloro – 2 (3H) – benzoxazolinone – 3 – acetyl – 2 – (p – dimethylaminobenzaldehyde) – hydrazone were significantly active than phenytoin (a commercial antiepileptic drug) in the tests.

Hydrozones in addition to Schiff and Mannich bases of isatin were evaluated for anticonvulsant activity by maximal electroshock method (MES) and metrazol-induced convulsions (MET) at different dose levels. Neurotoxicity of the compounds was also noticed at the same dose levels. Eight compounds of the series denoted significant anticonvulsant activity at 30 mg/kg dose level. 3-(4-chloro-phenylimino)-5 methyl-1.3-dihydro-indol-2 one showed to be the most potent compound of the series with 87% protection at 100 mg/kg and an ED (50) of 53.61 mg/kg (MET).

Anti-inflammatory and Analgesic activity.

Non-steroidal anti-inflammatory drugs (NSAIDS) are largely used in the treatment of pain and inflammation, Hydrazones that are dual inhibitors of both cyclooxygenase (COX) and 5- lipooxygenase (5-LO) are being studied as potential analgesic and anti-inflammatory agents in comparison to NSAIDS.

Fifteen different isatin [N-(2-alkylbenzoxazole-5-carbonyl)] hydrazones were synthesized and screened for analgesic, antidepressant and H1-antihistaminic activities. These compounds were also studied for their effect on pentobarbitone – induced narcosis. Results revealed that three compounds bearing a methyl substituent at 7 – position of the benzoxazole system exhibits good analgesic activity, in relation to standard.



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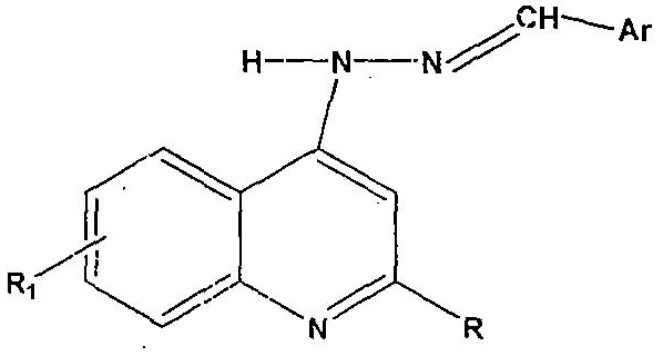
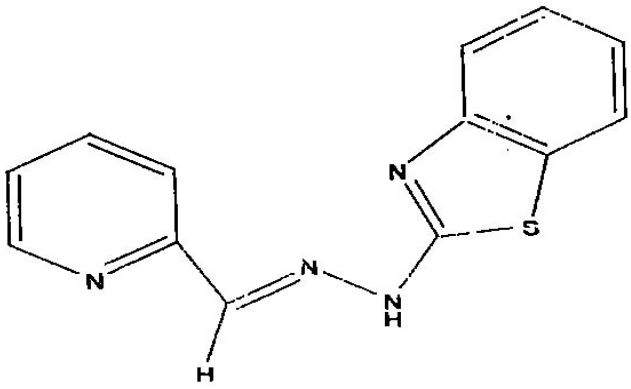
Antituberculosis activity

Tuberculosis (TB) is the leading single-agent infectious disease killer in the world. Time major challenges for tuberculosis control are the development of multidrug-resistant tuberculosis (MDRTB) strains. As a result, there is a pressing need for new antitubercular agents acting with greater potency and efficacy than the current existing drugs.



Antitumor activity

The search for antitumor drugs has led to the discover of several hydrazones having antitumor activities. Novel 2-benzimidazolyl- 2-benzoxazolyl- and 2-benzothiazolyl hydrazones 10 are synthesized from 2-formylpyridine, 2-acylpyridines, acetyldiazines and acetyl(iso) quinolines theses compounds exhibited potent cytotoxic and antitumor activities and are also useful against multidrug resistant cancer cells. The antiproliferative actively of the compounds has been tested in various tumor cell lines.



Antimalarial activity

Malaria is a major health problem in poverty-striken regions where new antiparasitic drugs are required at an affordable price. Malaria is caused by parasitic protozoa of the genus plasmodium. There is a need of intensive search for compounds having antimalarial activity against multi-drug resistant plasmodium falciparum.

A Walcourt et.al. investigated antimalarial activity of novel aroyl hydrazone and thisosemicarbazone Fe chelators. These compounds inhibited the growth of tumor cell lines in cell culture [Blood 100(2002) 666] suggesting them to be highly active. The most effective chelators examined were 2- hydroxyl – 1- napthaldehyde – 4 – phenyl – 3 – thiosemicarbazone.

A series of quinolylhydrazones were synthesized and their antimalarial activity was evaluated against the chloroquine-sensitive strain of Plasmodium falciparum. One of the compounds displayed an activity 6 fold higher than

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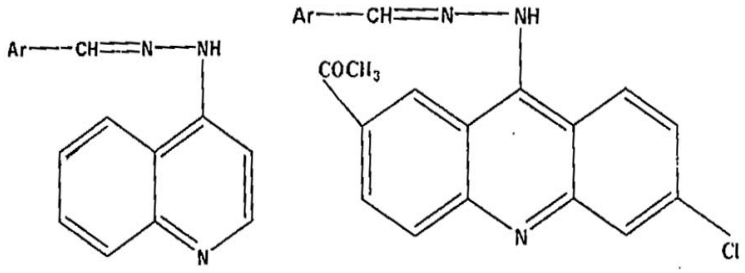
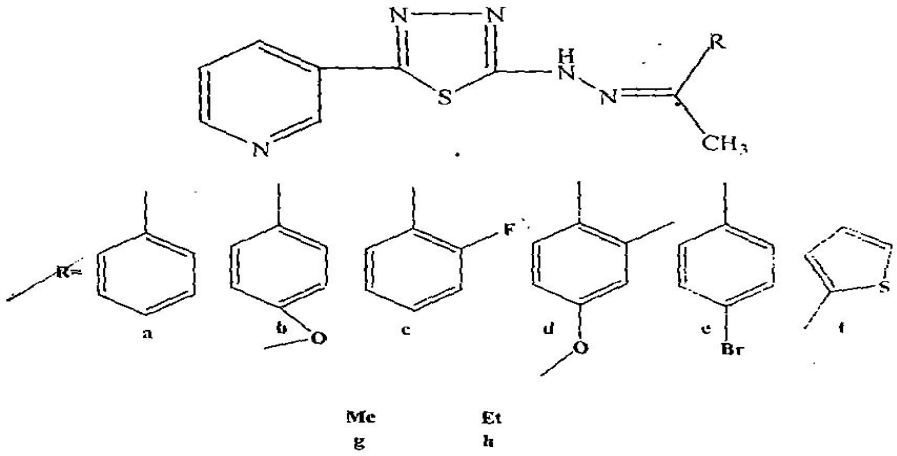
cloroquine (CQ) and none of the active compound was found to inhibit β – hematin formation in vitro in the same range as chloroquine.

A series of N1-arylidene – N2 – quinolyl and N2 – acrydinylhydrazones (16a, 16b) were synthesized and tested for their antimalarial properties. The synthesized compounds showed an antiplasmodial activity against the chloroquine-sensitive D10 strain in the same range of chloroquine (CQ)



Antioxidant activity.

K.J. Pratap et.al. synthesized a new series of Ketone 1-(5-(pyridine-3-yl)-1,3,4- thiadiazol-2-yl)-hydrazone derivatives (19a 19h) by the condensation of 1-(5-(pyridine-3-yl)-1,3,4- thiadiazol-2-yl)-hydrazone with substituted and unsubstituted ketones. They evaluated their antioxidant property by using 1, 1-diphenyl-2-picrylhydrazil (DPPH) method. All the compounds demonstrated good antioxidant activity due to the presence of (-NH-N=) moiety attached to aryl and heteroaryl nuclei thereby, stabilizing the free radical



IV. CONCLUSIONS

All the complexes contain lattice water and shows weight loss up to 320°C indicates decomposition of ligand molecule. Further a horizontal curve was observed beyond 640°C suggest the formation of final decomposition products i.e. stable metal oxides of respective metals. On the basis of half decomposition temperature the order of thermal stability is found to be Ni (II) > Cu (II) > Zn (11) > Co (II). The analysis of magnetic moment, thermal analysis and electronic spectral data shows structural changes. The structural changes of all complexes have marked effect on the sensitivity and sensitivity varies with organisms.

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