SYNTHESIS, CHARACTERIZATION AND ANTIOXIDANT STUDY OF SCHIFF BASES

Project work by

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MASTER OF SCIENCE IN

PHARMACEUTICAL CHEMISTRY

Under the Guidance of

BAIJU K.P

DEPARTMENT OF CHEMISTRY

BHARATA MATA COLLEGE, THRIKKAKARA

CERTIFICATE

This is to certify that this project work entitled **"Synthesis, characterization and antioxidant study of Schiff bases"** is an authentic record of the project work carried out by **KRISHNAPRIYA PB** of final MSc Chemistry under the supervision and guidance of **Dr. P. V. MOHANAN**, Professor of Department of Applied Chemistry, Cochin University of Science and Technology in the partial fulfillment of the requirement for the award of the degree of Master of Science in Pharmaceutical Chemistry of Bharata Mata college, Mahatma Gandhi university, Kottayam during the academic year 2018-2020.

Cochin-22

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CERTIFICATE

This is to certify that the project report entitled "**SYNTHESIS, CHARACTERIZATION AND ANTIOXIDANT STUDY OF SCHIFF BASES**" is an authentic record of the project work carried out by **Ms. KARISHNAPRIYA P.B** (Reg.no:180011017731) in partial fulfillment of the award of the degree of Master of Science in Pharmaceutical chemistry at Bharata Mata College, Thrikkakara affiliated to Mahatma Gandhi University, Kottayam under my guidance and supervision during 2018-2020.

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DECLARATION

I, KRISHNAPRIYA P.B, hereby declare that the work presented in this project entitled **Synthesis, characterization and antioxidant study of Schiff Bases** is entirely original work which was carried independently under the supervision of **Dr. P. V Mohanan**, Professor of Department of Applied Chemistry, Cochin University of Science and Technology and has not been included in any other project submitted previously for the award of any other degree.

Thrikkakara **KRISHNAPRIYA. P. B**

June 2020

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KRISHNAPRIYA. P.B.

CONTENTS

CHAPTER 1

INRTODUCTION

1.1 Schiff Base

The compounds containing the azomethine group (-CH= N-) are known as Schiff base. Schiff bases are the condensation products of primary amines and carbonyl compounds and they were discovered by a German Chemist, Nobel Prize winner Hugo Schiff in 1864[1].They have the general formula,

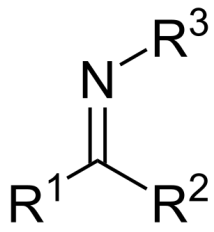


Figure: 1 **Structure of Schiff base ligand**

Where R^3 is an alkyl or aryl group (not a Hydrogen) which makes the Schiff base a stable imine and R^1 and R^2 may be Hydrogen [2].

Different synthetic strategies have been reported for the preparation of Schiff bases like solid-state synthesis, using dehydrating agents, silica/ultrasound irradiation or microwave assisted synthesis [3].

The classical method involves the condensation of an aldehyde and a primary amine under acidic or basic conditions [4].

The mechanism of formation of a Schiff base from an aldehyde or ketone is a reversible reaction and generally takes place under acidic or basic catalysis or upon heating. The formation generally driven to the completion by separation of the product or removal of water or both. ManySchiff bases can be hydrolyzed back to their aldehydes or ketones and amines by aqueous acid or base [5].

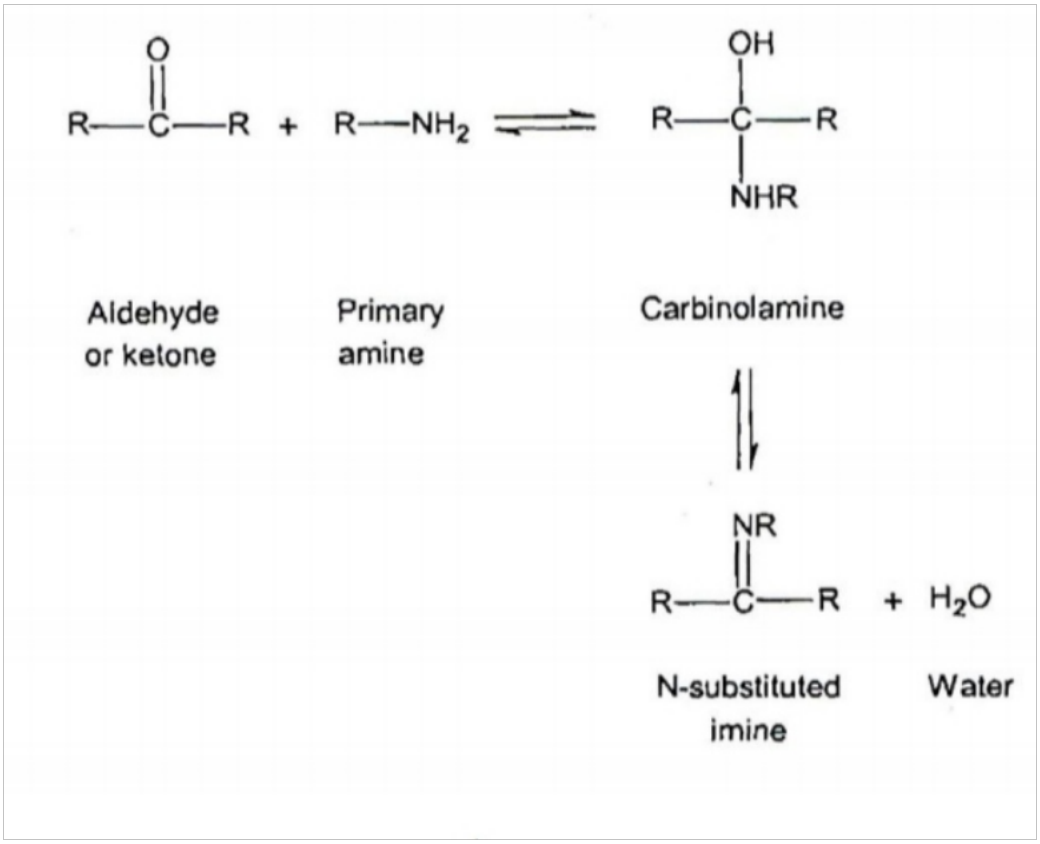


Figure: 2 Schiff Base Reaction

The dehydration of carbinol amine is also catalyzed by base. This reaction is somewhat analogous to the E_2 elimination of alkyl halides expect that it is not a concerted reaction. It proceeds in two steps through an anionic intermediate. The Schiff base formation is really a sequence of two types of reaction i.e.; addition followed by elimination. Yet, the acid concentration cannot be too high because amines are basic compounds. If the amines is protonated and becomes non-nucleophile, equilibrium is pulled to the left and carbinol amine formation cannot occur. Therefore, many Schiff base synthesis are best carried out at mild acidic pH.

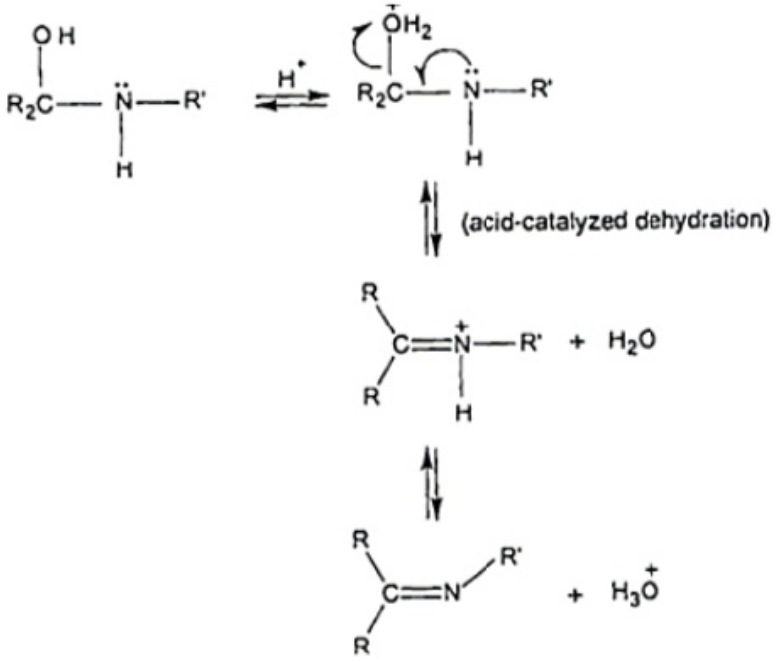


Figure: 3 General scheme for the formation of Schiff base

The mechanism of Schiff base formation is another variation on the theme of nucleophilic addition to the carbonyl group. In this case, the nucleophile is the amine. In the first part of the mechanism, the amine reacts with the aldehyde or ketone to give an unstable addition compound called carbinol amine. The carbinol amine loses water by either acid or base catalyzed pathways. Since the carbinol amine is an alcohol, it undergoes acid catalysed dehydration [6].

1.2 Application of Schiff base ligands.

Schiff bases are Aldehydes or ketone compounds where the carbonyl group is replaced by an azomethine or imine group. Schiff bases form an important class of the most widely used organic compounds and have wide variety of applications in many fields including analytical, biological and inorganic chemistry. Schiff bases gained importance in medicinal and pharmaceutical fields due to a broad spectrum of biological activities like anti-inflammatory, analgesic, antimicrobial,anticonvulsant,and antitubercular,anticancer,antioxidant,anthelmintic and so forth. Schiff bases are also used as catalyst, intermediates in organic

synthesis, dyes, pigments, polymer stabilizers, and corrosion inhibitors. Studies enlightened that metal complexes show greater biological activity than free organic compounds. Augmentation of biological activity was reported by implementation of transition metals into Schiff bases.

Schiff bases played an influencing role in development of coordination chemistry and were involved as key point in the development of inorganic biochemistry and optical material.Schiiff bases have been utilized as synthons in the preparation of a number of industrial and biologically active compounds like formazans, 4-thiazolidines, benzoxazines and so forth, via ring closure, cycloaddition and replacement reactions .Schiff base derivatives in various processes promoted the researchers for designing of novel heterocyclic/ aryl Schiff bases for development of new environmental- friendly technology[7].

1.2.1 Schiff base as a corrosion inhibitor

One of the important applications of Schiff base is their use as an effective corrosion inhibitor, which is based on their ability to spontaneously form a monolayer on the surface to be protected. The principle interaction between inhibitor and metal surface is chemisorption [8].The inhibitor molecules have centres capable of forming bonds with the metal surface by electron transfer. In such cases the metal acts as an electrophile and inhibitor acts as Lewis base. Nucleophilic centres, such as oxygen and nitrogen atoms, of the protective compound have free electron pairs which are readily available for sharing. Together with the atoms of benzene rings they create multiple absorption sites for the inhibitor thus enabling stable monolayerformation [9].

1.2.2 Schiff Base ligands used as dyes

Schiff bases derived from aniline moietycontaining phenyl or substituted phenyl groups, which sometimes called Azo dyes. These Schiff bases can be directly prepared from aromatic amines with aromatic carbonyl groups, which are stable and can be manipulated understand different suitable conditions. The phenomenon of coordination of Schiff base with metal ions give the Schiff bases the good advantages to be introduced in the dye synthesis[10].Some metal complexes [11] are

used to mass dye polyfibers. Cobalt complex of a Schiff base (salicylaldehyde with diamine) has excellent light resistance and storage ability and does not degrade even in acidic gases (CO_2). Novel tetradentate Schiff base acts as a chromogenic reagent for determination of Ni in some natural food samples.

1.3 Biological activity of Schiff base ligands

1.3.1 Antioxidant activity

Free radicals are produced under certain environmental conditions and during normal cellular functions in the body. Antioxidants thus play an important role to protect the human body against damage by reactive oxygen species. The ability of Schiff bases and their metal complexes to scavenge free radicals is an important property. Presently a number of Schiff base metal complexes have been investigated as antioxidants. A new series of multipotent antioxidants (MPAOs), named Schiff base 1,2,4- triazoles attached to the oxygen- derived free radical scavenging moiety butylated hydroxytoluene (BHT) we're synthesised by K. T Thadele and their antioxidant activity were evaluated by 2,2-diphenyl- 1- picryl hydrazyl (DPPH) method[12].

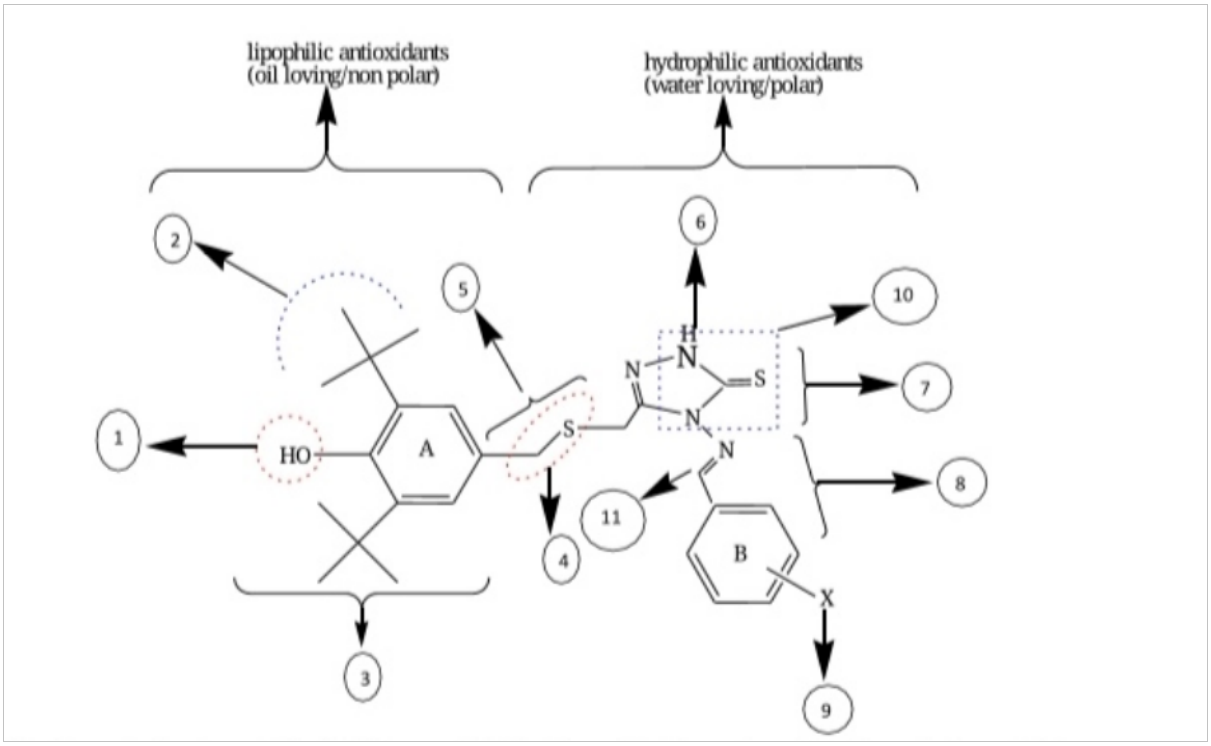


Figure: 4

Presently, synthetic antioxidants are widely used because theyare effective and cheaper than natural antioxidants. Currently a number of Schiff – base metal complexes have been investigated as effective scavengers of ROS, acting as antioxidants.

1.3.2 Anticonvulsant activity

A Novel Schiff bases of isatin were synthesized by condensation of imesatin with different aromatic aldehydes. The imesatin were synthesized by reaction of isatin with p- phenylenediamine. All the synthesized compounds screened for anticonvulsant activities against maximal electroshock (MES) and subcutaneous metrazole (ScMet). Among the compounds synthesized 3-(4-(3, 4,5- trimethoxy benzylideneamino) phenylimino) indoline-2- one showed excellent anticonvulsant activity with lower dose in MES as well as in ScMet methods. Thus compound may be chosen as a prototype for development of new anticonvulsants [13].

Among the important pharmacophores responsible for anticonvulsant activity, the isatin scaffold is stills considered a viable lead structure for the synthesis of more efficacious anticonvulsant activity. Isatin was reported to possess proconvulsant and anticonvulsant activities apart from other pharmacological properties. It was envisaged that Schiff bases of isatin would also exhibit significant anticonvulsant activity; we hereby report the anticonvulsant activity of Schiff bases of isatin by maximal electroshock method (MES) and metrazol- induced convulsions (MET). The neurotoxicity of the compounds was also assessed for the compounds at the experimental dose levels.

1.3.3 Antimicrobial activity

Sonu et al have synthesized the superior antimicrobial compounds with different substituted aromatic aldehydes/acetophenones are selected as the starting material for the synthesis of Schiff base by means of sulphonamide assists to formation of Schiff base in presence of alcohol and acidic reagent. All the synthesized Schiff bases of sulphonamide have revealed excellent antimicrobial activity.

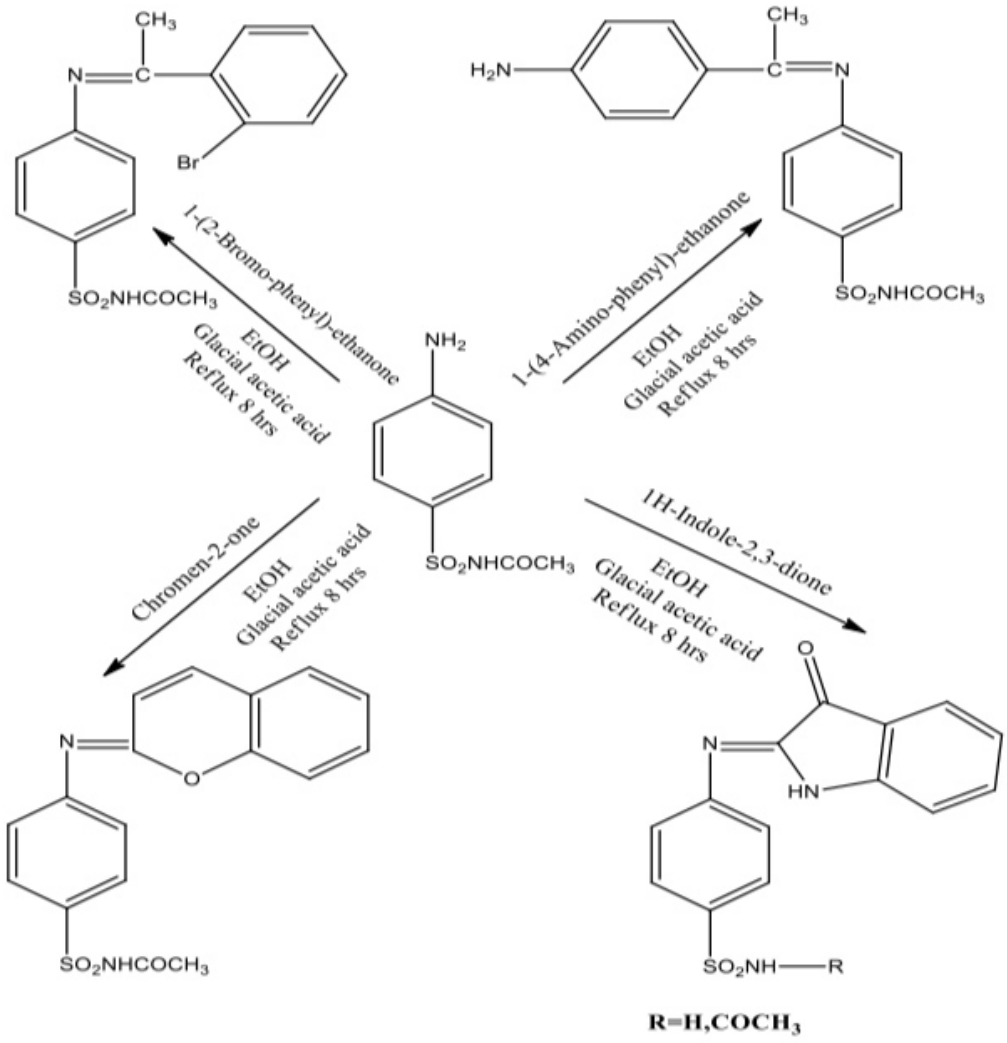


Figure: 5 Sulphonamide Derivatives of Schiff bases

1.3.4 Antibacterial Activity

Aysen et al have reported the synthesis characterization and investigation of antibacterial activities of five bacteria of Schiff base ligands. From 4-benzyloxybenzaldehyde and 2-aminopyridine, (4-benzyloxy-benzylidine)pyridine-2-yl

amine was derived.

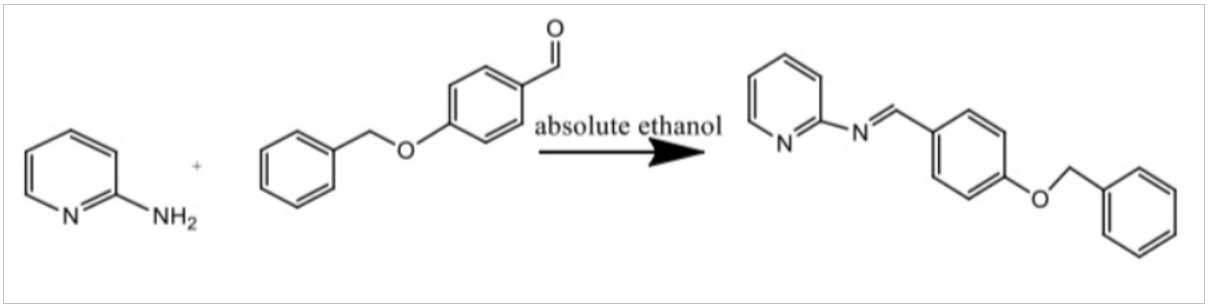


Figure: 6

AK Kaura et al reported the synthesis of some novel Schiff base of biological importance. With the help of Agarwell diffusion method all the compounds have been screened for their invitro antibacterial acitivity against gram positive and gram negative bacterial strains. From this, the given compound (1) show maximum activity against Bacillus subtilies.

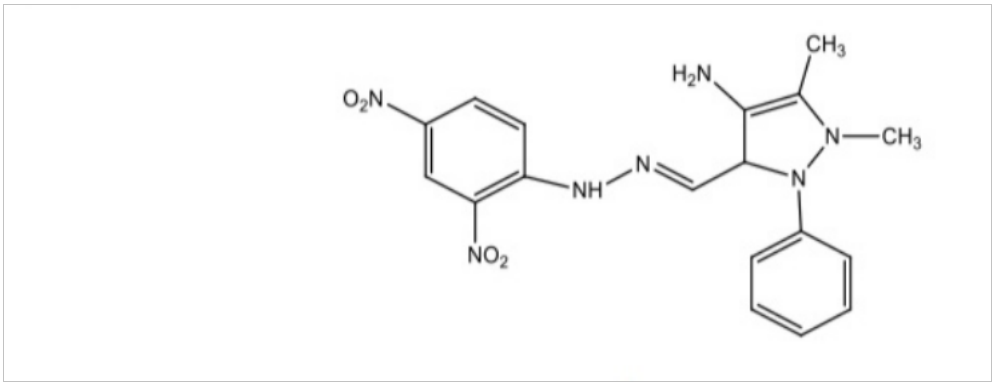


Figure:7 Compound(1)

1.4 Importance of 2-amino-4-methylphenol, syringaldehyde, 4-aminoantipyrene, 2,5-dimethyl benzaldehyde.

2-amino-4-methylphenol is the major sensitizer in contact allergy to disperse yellow3. It reacts with acetyl acetone in absolute ethanol to yield 4-(2-hydroxy-5-methylphenyl) imino-2-pentanone. It was converted to dihydrophenoxazinone by purified human haemoglobin. 2-amino-4-methylphenol was used in the synthesis of novel functionalized spiropyran derivatives of 2H-1,3,benzoxazinone series.

Syringaldehyde is a promising aromatic aldehyde which possess worthy bioactive

properties and is therefore, used in pharmaceuticals, food, cosmetics, paper industrial and even in biological control applications. Mostly the synthetic form of syringaldehyde is being used. Syringaldehyde or 3,5dimethoxy-4-hydroxybenzaldehyde is a naturally occurring unique compound with assorted bioactive characteristics belongs to the phenolic aldehyde family. It is similar in structure to its infamous counterpart, vanillin and it has comparable applications [16].

4–aminoantipyrene based heterocyclic's have gained great importance as it is abundant in nature and wide pharmacological activities [17].It also possess potential diverse applications in biological, clinical and analytical areas [18].4-aminoantipyrene is a pyrazole derivative which has antipyretic action [19].It is used in the preparation of azodyes [20].4- aminoantipyrene is also used to protect against oxidative stress as well as prophylactic of certain diseases including cancer.

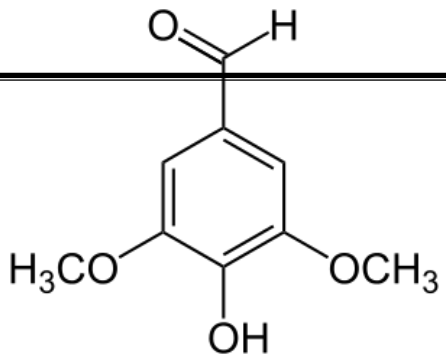


Figure:8 2-amino-4-methylphenol

Figure:9 Syringaldehyde

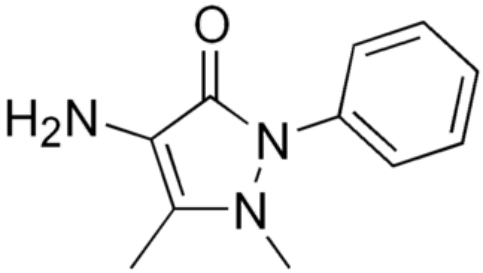


Figure:10 4-aminoantipyrene

1.5 SCOPE OF PRESENT INVESTIGATION

Schiff bases are considered as very important organic compounds because of their ability to form complexes with transition metal ions and its pharmacological properties. Because of its various applications in biological process and potential application of designing new therapeutic agents. Many Schiff bases were found to satisfy the requirements for acting as catalyst for oxidation of organic substances. Selective oxidations of organic substances are important in chemical and photochemical industries due to the wide variety of products synthesized in this route. Many Schiff base complexes have antioxidant, anticancerous, antibacterial activities and so on. The wide applicability of these compounds inspired us to synthesize new Schiff base compounds.

CHAPTER II

MATERIALS AND METHODS

2.1 REAGENTS

1) Syringaldehyde

2) 2-amino 4-methyl phenol

3) 4-amino antipyrene

4) 2,5 dimethyl benzaldehyde

2.2 SOLVENTS

1) Methanol

2) Ethanol

3) DMF

4) DMSO

5) $CHCl_3$

2.3 INSTRUMENTAL TECHNIQUES

2.3.1 ELEMENTAL ANALYSIS

Elemental analysis is a process in which sample of some materials is analysed for its elemental composition. Elemental analysis can be quantitative and qualitative. The most common form of elemental analysis is CHN analysis which was done on a Vario EL IIICHN elemental analyzer at the SAIF, Cochin University of Science And Technology, Kochi, India

2.3.2 INFRARED SPECTROSCOPY

Infrared spectroscopy (IR spectroscopy) is the spectroscopy that deals with the infrared region of the electromagnetic spectrum that is light with a longer wavelength and lower frequency than visible light. It covers a range of techniques, mostly based on absorption spectroscopy. It is used to identify the structure since the functional groups give rise to characteristic bands both in terms of intensity and position. It is based on the vibrations of atoms of a molecule. A common laboratory instrument that uses this techniques is a Fourier transform infrared (FTIR) spectrometer. Powders, being examined by infrared spectroscopy, in transmission are generally prepared by grinding with potassium bromide (KBr) powder. The latter is then pressed into a disk.

Infrared spectra were recorded on a JASCO FT-IR spectrometer in the range 4000-400 cm-1 using KBr pellets at Department of Applied Chemistry, Cochin University of Science And Technology, Kochi, India.

2.3.3 ELECTRONIC SPECTROSCOPY

Electronic spectroscopy or ultraviolet-visible spectrophotometry (UV-Vis) refers to absorption spectroscopy in part of the ultraviolet and the full, adjacent visible spectral regions. In this regions of the electromagnetic spectrum, atoms and molecules undergo electronic transitions. Absorption spectroscopy is complementary to fluorescence spectroscopy, in that fluorescence deals with transition from the excited state to the ground state, while absorption measures transitions from the ground state to the excited state. Molecules containing bonding and non – bonding electrons (n- electrons) can absorb energy in the form of ultraviolet or visible light to excite these electrons to higher anti-bonding molecular orbitals. The more easily excited the electrons (i.e. lower energy gap between the HOMO and the LUMO), the longer the wavelength of light it can absorb. There are four possible types of transitions (n → π^*, $\pi \rightarrow \pi^*$, $\sigma \rightarrow \sigma^*$ and n → σ^*) and they can be ordered as follows in terms of energy; $\sigma \rightarrow \sigma^*$> n → σ^*>$\pi \rightarrow \pi^*$> n → π^*.

Electronic spectra were recorded in DMF on a SPECTRO UV visible double beam UVD

-3500 spectrometer in the range 200 to 900 at the Department Of Applied Chemistry, Cochin University of Science And Technology, Kochi, India.

2.3.4 NMR SPECTROSCOPY

NMR is a Spectroscopic method that is even more important to the chemists than infrared spectroscopy. Many nuclei may be studied by NMR technique, but hydrogen and carbon are most commonly available. Whereas infrared spectroscopy reveals the type of functional group present in a molecule. NMR gives the information about the number of magnetically distinct atom of the type being studied. NMR involves in the interaction between an oscillating magnetic field of electromagnetic radiation and the magnetic energy of the hydrogen nucleus or some other type of nuclei are placed in an external magnetic field. The sample absorbs electromagnetic radiation in radio wave region at different frequencies since absorption depends upon the type of proton or certain nuclei contained in the sample.

This technique consist in exposing the proton (placing the substance) in an organic molecule to a powerful external field. The protons will press at different frequencies. Now we irradiate the pressing protons with steadily changing frequencies. It is generally convenient to keep the radio frequency constant and the strength of the magnetic field is constantly varied. At some value of the field strength, the energy required to flip the proton matches the energy of the radiation. Absorption occurs and a signal is observed. In NMR spectrum, we measure applied field strength for each set of protons and the absorption peak are plotted.

'H NMR spectra are recorded in chloroform on a Bruker Advance 111400 MHz FT-NMR spectrometer using TMS as the internal standard at SAIF, Cochin University of Science and Technology, Kochi, India.

2.3.5 MASS SPECTROSCOPY

Mass spectroscopy (MS) is an analytical technique that measures the mass-to-charge ratio of an ions. The results are typically presented as a mass spectrum, a

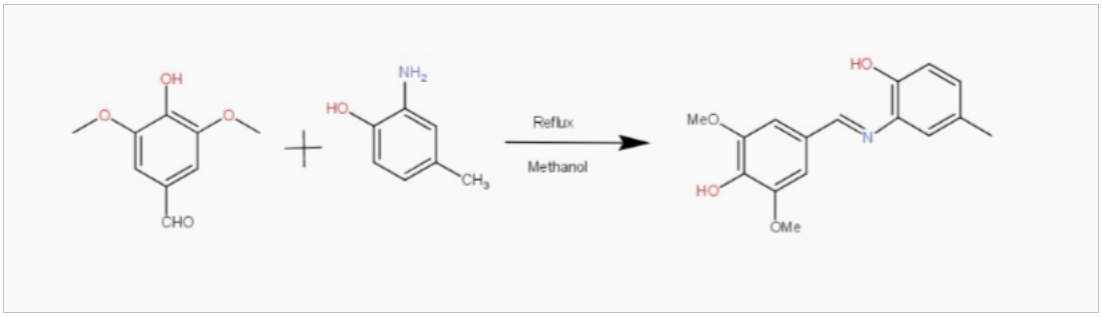
plot of intensity as a function of mass-to-charge ratio. Mass spectrometry is used in many different fields and is applied to pure samples as well as complex mixtures. A mass spectrum is a plot of the ion signal as a function of the mass to charge ratio. These spectra areused to determine the elemental or isotopic signature of a sample, the masses of particles and of molecules, and to elucidate the chemical identity or structure of molecules and other chemical compounds. In a typical MS procedure, a sample, which may be solid, liquid or gas, is ionized, for example by bombarding it with electrons. This may cause some of the samples molecules to break into charged fragments or simply become charged without fragmenting. These ions are then separated according to their mass-to-charge ratio, for example by accelerating them and subjecting them to an electric or magnetic field: ions of the same mass-to-charge ratio will undergo the same amount of deflection. The ions are detected by a mechanism capable of detecting charged particles, such as an electron multiplier. Results are displayed as spectra of the signal intensity of detected ions as a function of mass-to-charge ratio. The atoms or molecules in the sample can be identified by correlating known masses (e.g. an entire molecule) to the identified masses or through a characteristic fragmentation.

The instrument used for Mass spectrometry is waters 3100 Mass detector using ESI technique designed for routine LC-MS analysis was recorded at Department of Applied Chemistry, CUSAT.

2.4 PREPARATION OF LIGANDS

2.4.1 Synthesis of Schiff base from Syringaldehyde and 2-amino 4-methyl phenol (A)

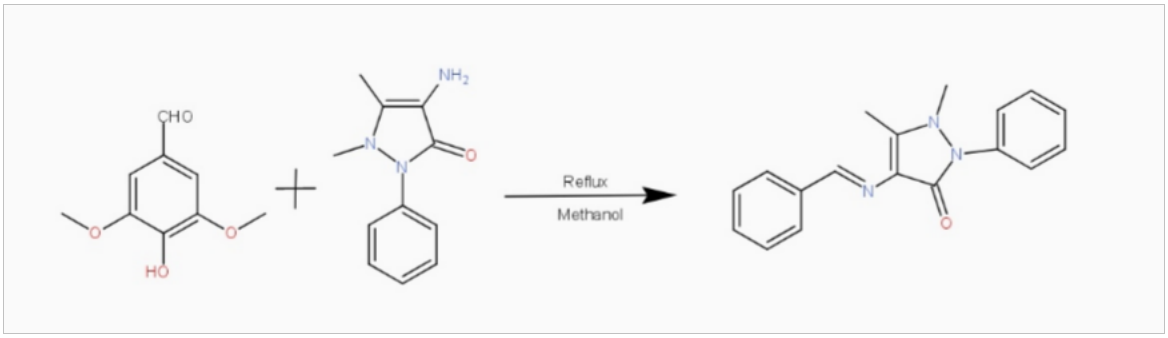
4-((2-hydroxy-5-methylphenyliimino) methyl) -2, 6-dimethoxy phenol was synthesized from the methanolic solution of Syringaldehyde (8 mmol) and methanolic solution of and 2-amino-4-methyl phenol (8 mmol). The mixture was refluxed on a water bath for 5-6 h. The solvent evaporated slowly. Crystalline products are obtained were filtered, washed with water, methanol and ether and recrystallized twice from ethanol methanol mixture. The yield and melting point of the product were determined. It is soluble in methanol, ethanol, DMF, DMSO, $CHCl_3$



Scheme: 1 Preparation of compound A

2.4.2 Synthesis of Schiff base from Syringaldehyde and 4-aminoantipyrene (B)

4-hydroxy3,5dimethoxybenzylideneamino)-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one was synthesized from methanolic solution of Syringaldehyde (8mmol) and methanolic solution of 4-amino antipyrene (8 mmol). The mixture was refluxed on a boiling water bath for 5-6 h. The solvent evaporated slowly. Crystalline products obtained were filtered, washed with water, methanol and ether and recrystallized twice from ethanol methanol mixture. The yield and melting point of the product were determined. It is soluble in $CHCl_3$, DMF and DMSO and slightly soluble in methanol and ethanol.

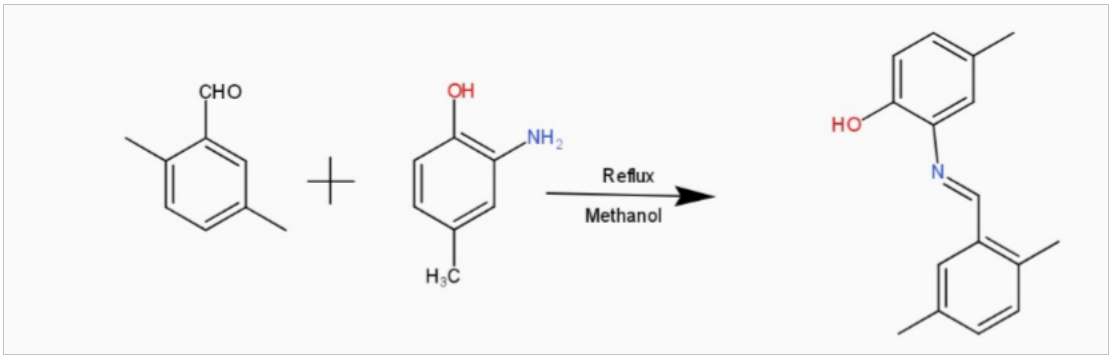


Scheme: 2 preparation of compound B

2.4.3 Synthesis of Schiff base from 2,5-dimethyl benzaldehyde and 2-amino 4-methyl phenol (C)

2-(2, 5 dimethylbenzylideneamino) -4-methyl was synthesized from the methanolic

solution of 2,5-dimethyl benzaldehyde and methanolic solution of 2-amino 4- methyl phenol. The mixture was refluxed on a boiling water bath for 5-6 h. The solvent evaporated slowly. Crystalline products obtained were filtered washed with water, methanol and ether and recrystallized twice from ethanol methanol mixture. The yield and melting point of the product were determined. It is soluble in methanol, ethanol, $CHCl_3$, DMF and DMSO.



Scheme: 3 Preparation of compound C

CHAPTER III

RESULTS AND CONCLUSIONS

3.1 ELEMENTAL ANALYSIS

Elemental analysis obtained is in good agreement with the assigned chemical formula of the proposed structure of ligands. The analytical data for the A, Band Care given the Table: 1

Table: 1 Elemental analysis

Compound	Empirical	Formula	Color	Calculated (found %)

	formula	Weight		C	H	N
A	$C_{16}H_{17}NO_4$	288.04	Yellow	66.18 (66.89)	5.32 (5.96)	4.67 (4.88)
B	$C_{20}H_{21}N_3O_4$	368.11	Orange	65.17 (65.38)	5.25 (5.76)	11.25 (11.44)
C	$C_{16}H_{17}NO$	240.12	Yellow	79.9 (80.30)	7.09 (7.16)	5.35 (6.69)

3.2 INFRARED SPECTROSCOPY

The IR bands of Schiff bases A, B and Cgive important information about the various functional groups present in it. The band shows in the range 1630-1500cm^{-1} which shows strong band of azomethine υ(HC=N) group. The IR spectra of compounds shows weak bands in the range 3500-3400cm^{-1} which indicate the phenolic OH group. FT-IR spectral bands of Schiff bases and their spectra are given in the Table: 2

Table: 2 IR spectrum of Schiff bases

Compound	υ(OH)	υ(C=N)	υ(CO)

A	3420	1501	1125 1310
B	3420	1615	1150
C	3413	1623	1035 1236

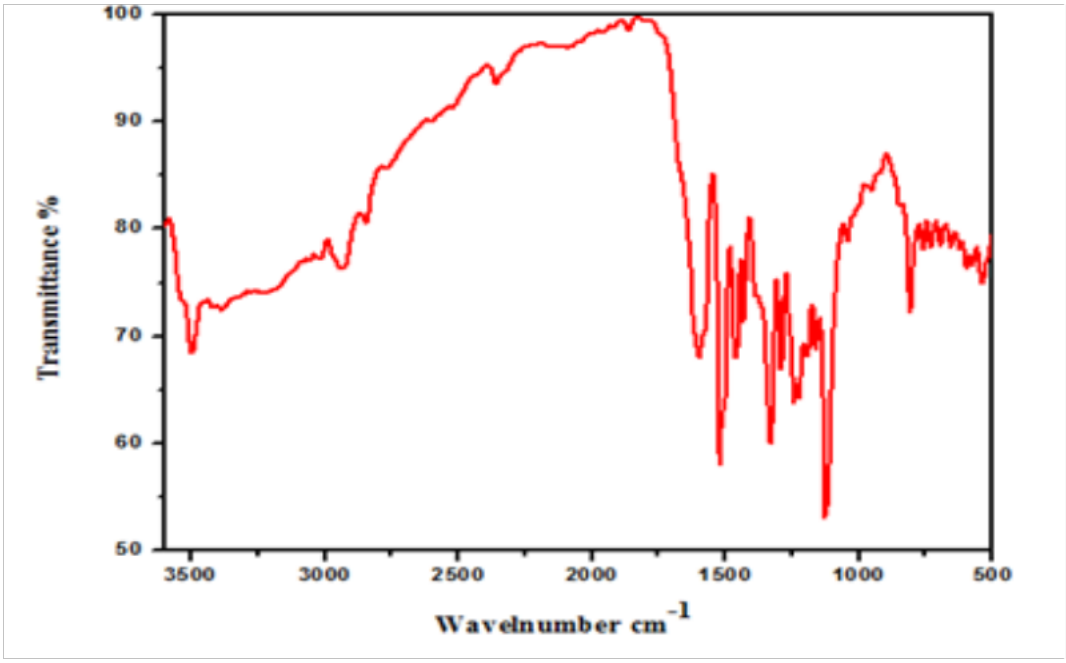


Figure: 11 IR spectrum of compound A

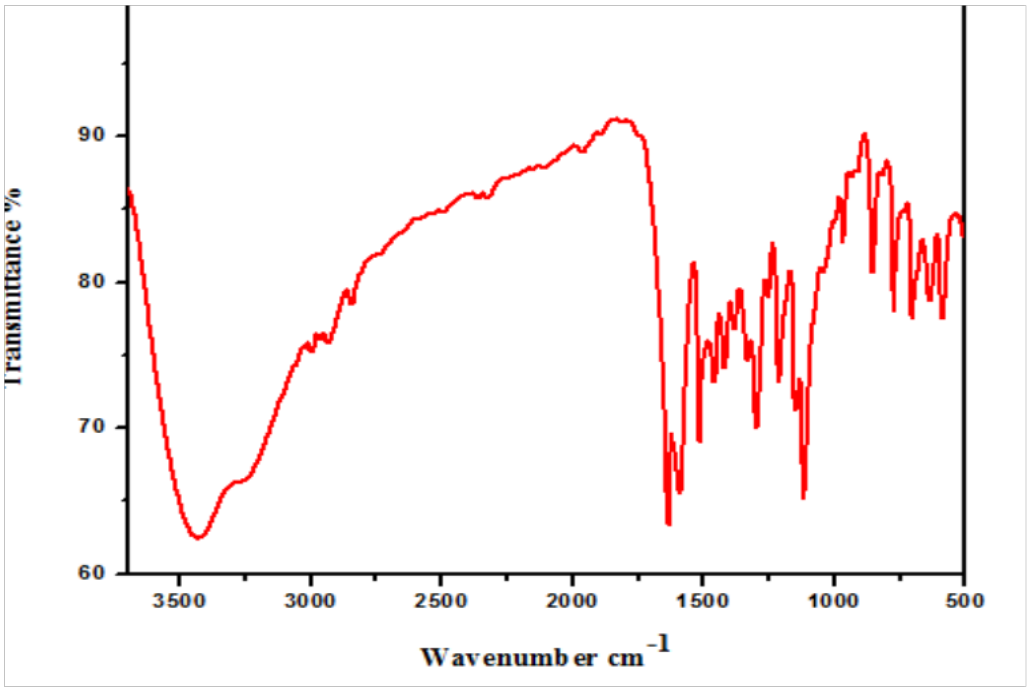


Figure: 12 IR spectrum of compound B

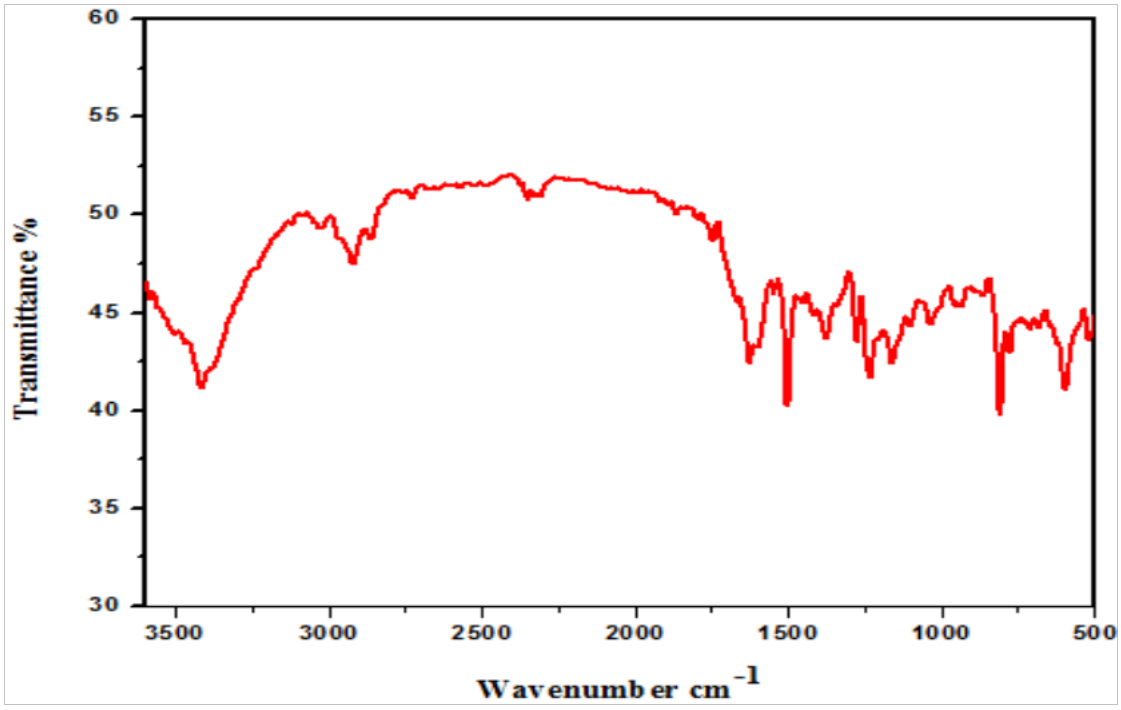


Figure: 13 IR spectrum of compound C

3.3 NMR SPECTROSCOPY

For unknown compounds, NMR can either be used to match against spectral libraries or to infer the basic structure directly. Once the basic structure is known, NMR can be used to determine molecular conformation in solution as well as studying physical properties at the molecular level such as conformational exchange, phase changes, solubility and diffusion. The singlet δ 8.4ppm correspondsto the azomethine proton (-CH=N-). The peaks at δ6.2ppm –δ7.1ppm corresponds to the aromatic protons. The broadness of the spectrum was due to the strong hydrogen bonding.

The ^{1}H NMR of the A ,B and C are recorded in $CHCl_3$ using TMS as internal standard which is shown in figures: 14,15 and 16.

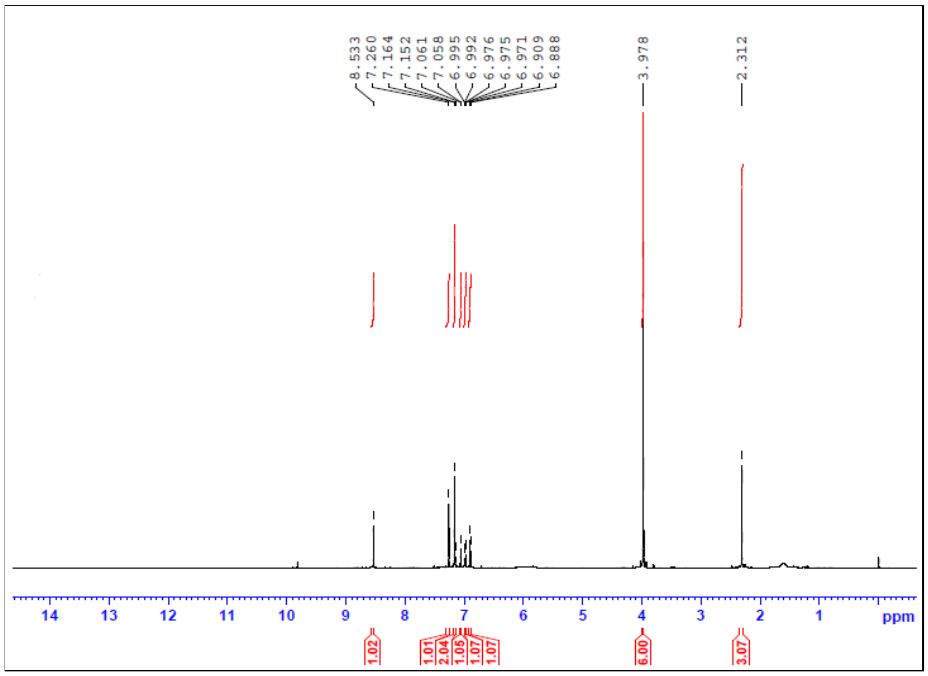


Figure: 14 ^{1}H-NMR of A

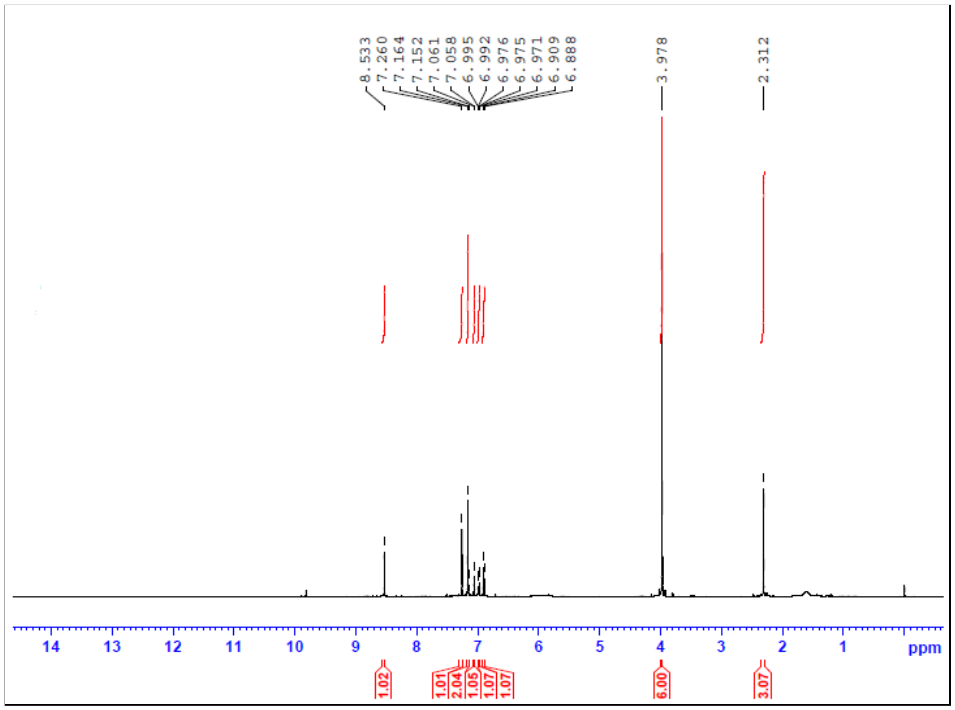


Figure:15 ^{1}H-NMR of B

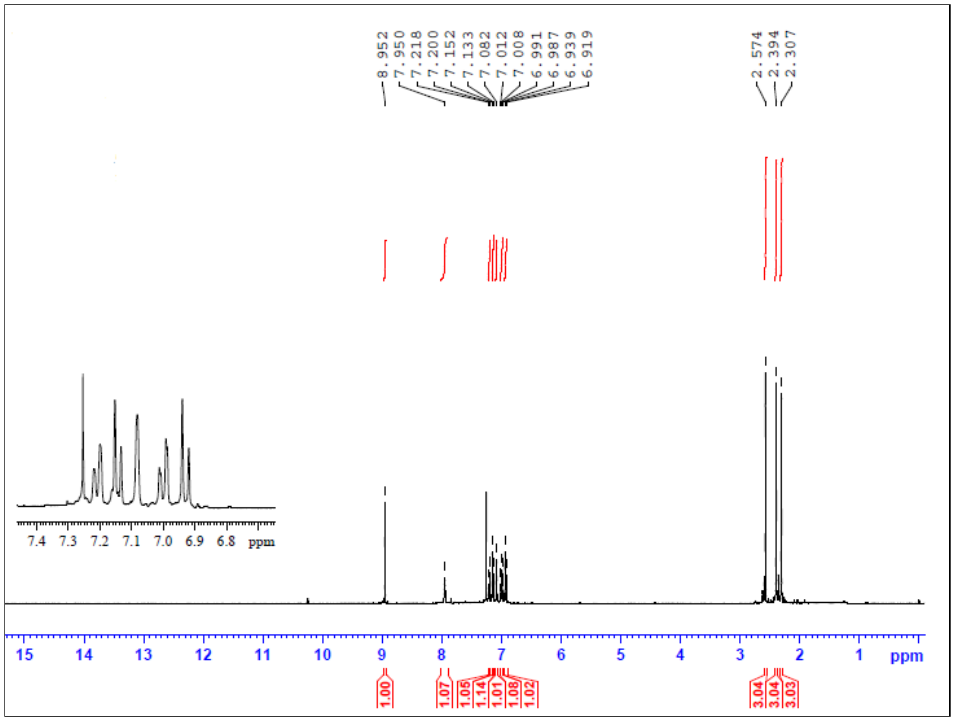


Figure: 16 ^{1}H-NMR of C

3.4 MASS SPECTRUM

Mass spectra is an accurate method used for determining the molecular mass of compound and its elemental analysis. Mass spectra (fig: 17, 18 and 19) shows molecular weight of the ligands A, B and C and stable peak are obtained are 288.04, 368.11 and 240.12.

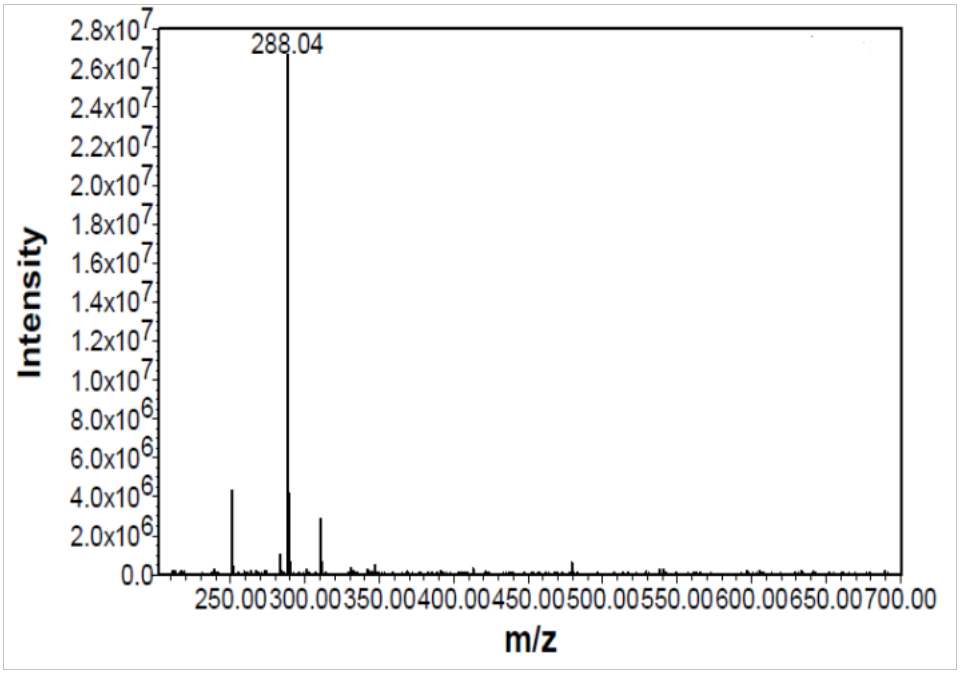


Figure: 17 Mass spectrum of A

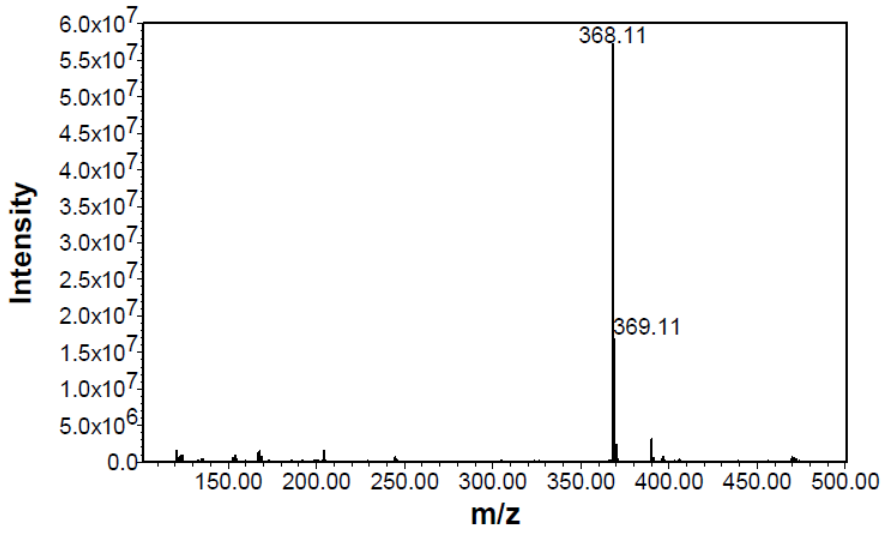


Figure: 18 Mass spectrum of B

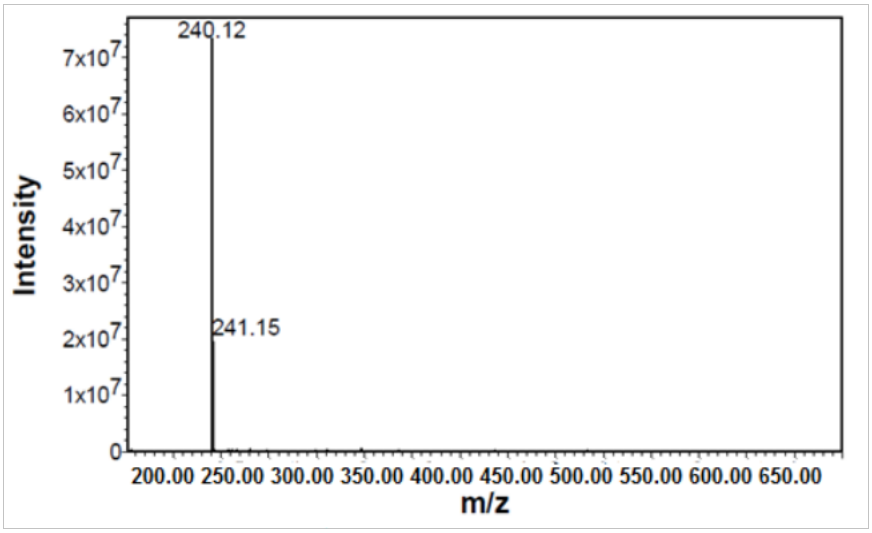


Figure: 19 Mass spectrum of C

3.5 UV-VISIBLE SPETROSCOPY

Electronic spectra are used to study the electronic structures and its dynamics in atom and molecules. The data are provided in Table 3. The electronic spectra of A, B and C were taken in DMSO. Transition within the aromatic ring assigned to π- π* transition. Transition within the C=N group assigned to be n-π* transition. The UV-Visible spectrum of A, B and C(fig: 20, 21 and 22) shows a characteristic band at 317nm, 350nm and 250nm respectively which indicates the π- π* transition within the aromatic ring. The bands of A and C at 360nm and 294nm signifies n-π* transition within azomethine group.

Table: 3 Electronic spectral data of ligands in DMSO

Compound	$\pi \rightarrow \pi^*$	$n \rightarrow \pi^*$
A	317	360
B	350	-

C	250	294

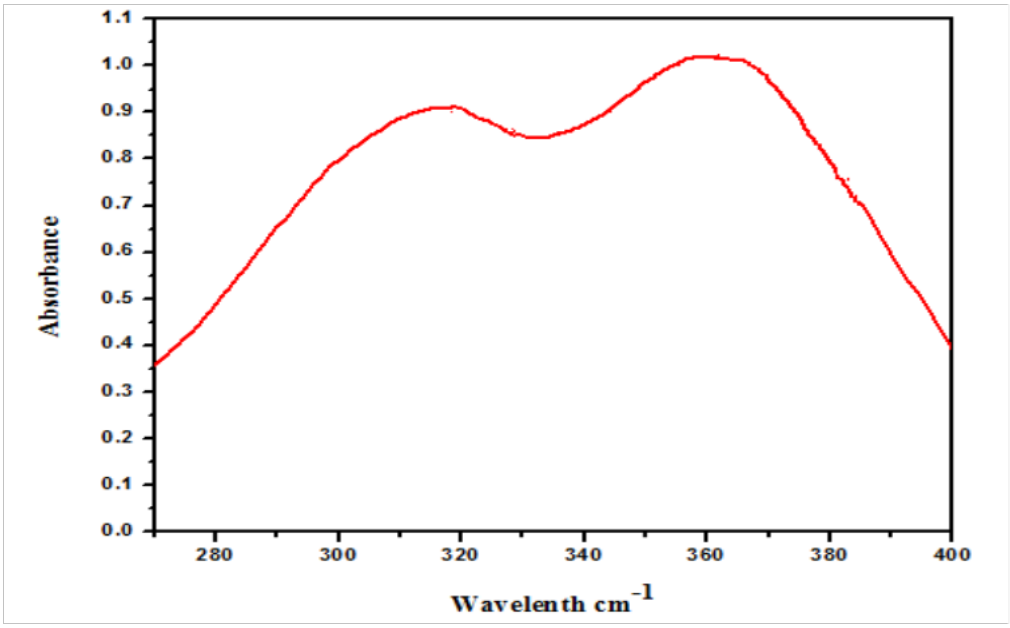


Figure: 20 electronic spectrum of A

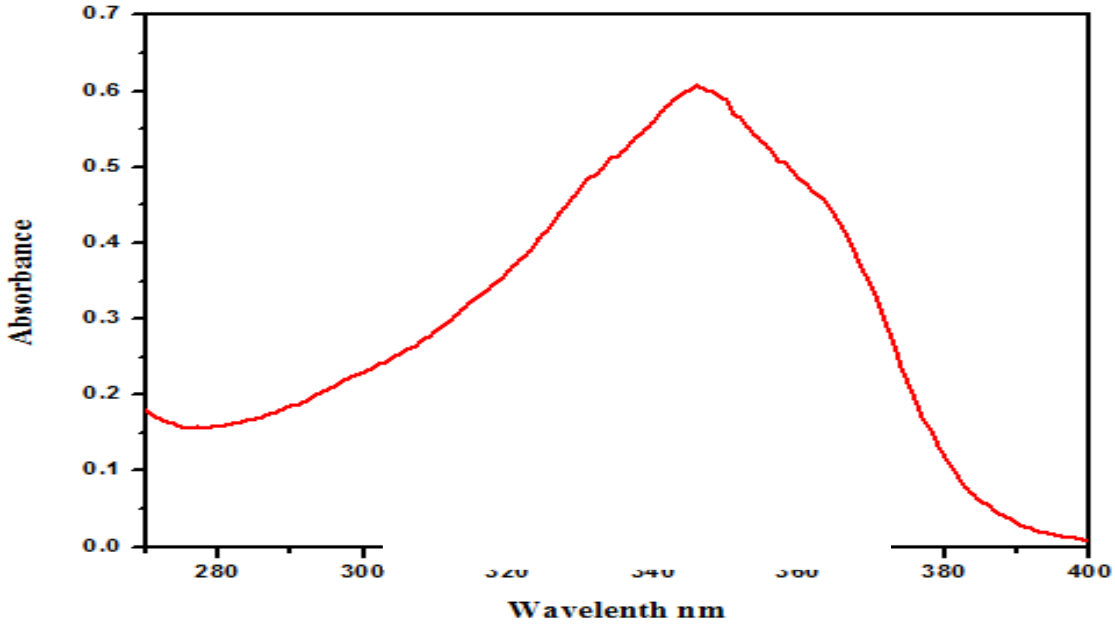


Figure: 21 electronic spectrum of B

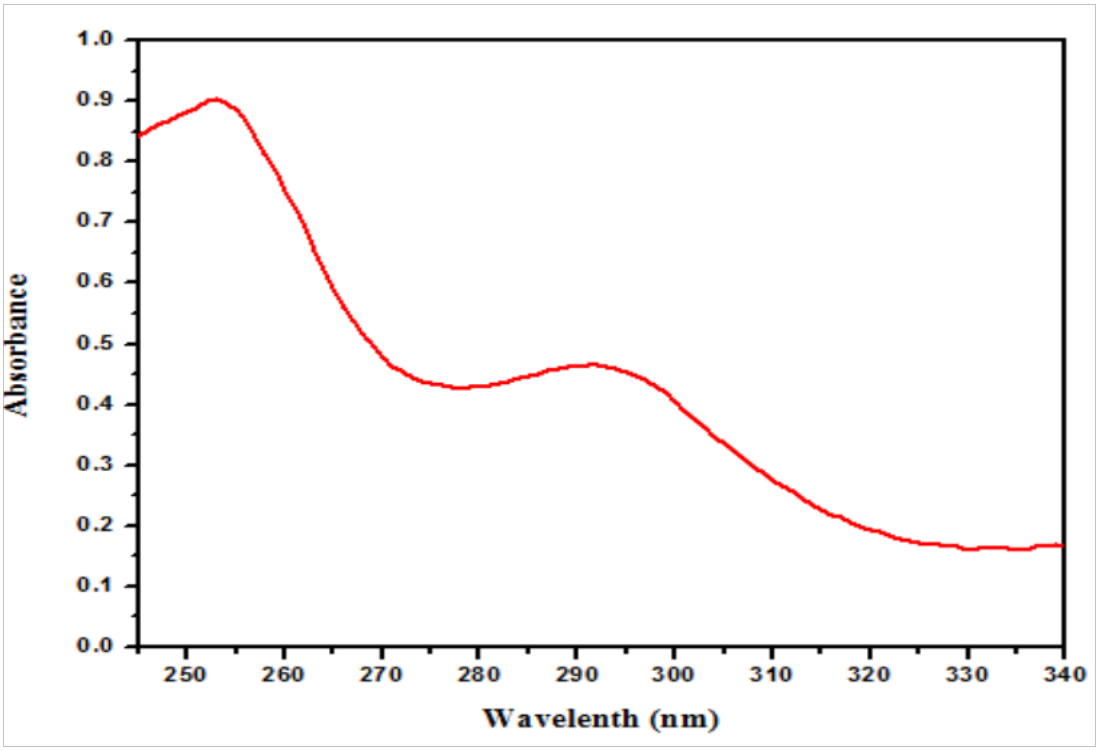


Figure: 22 electronic spectrum of C

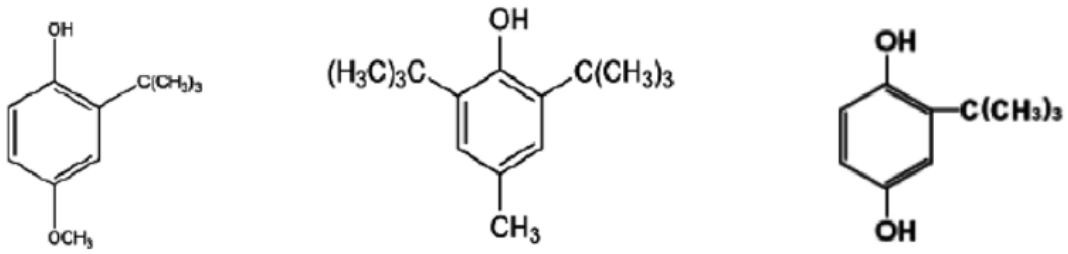
CHAPTER IV

ANTIOXIDANT ACTIVITY OF SCHIFF BASES

4.1 Antioxidant activity

Antioxidants are compounds which slow down or prevent the oxidation of other target molecules. They mop up free radicals and prevent them from causing cell damage.

Antioxidants are classified into natural, synthetic and nature identical antioxidants. Natural antioxidants are synthesized by microorganisms, fungi, animals and plants. Natural antioxidants are widely distributed in food and medicinal plants. These natural antioxidants, especially polyphenols and carotenoids, exhibit a wide range of biological 4effects, including anti-inflammatory, anti-aging, anti-atherosclerosis and anticancer. And the antioxidants identical to natural antioxidants but synthesized in the industry are called nature identical antioxidants. Antioxidants synthesized or biosynthesized by human are called synthetic antioxidants. Some examples of synthetic antioxidants are given bellow [21].



BHA BHT TBHQ

Figure: 23 Examples of synthetic antioxidants

4.2 Experiment

4.2.1 Materials

1. DPPH (2, 2- Diphenyl -1-picryl- hydrazyl)

2. Methanol

4.2.2 Methods

The antioxidant assay-radical scavenging activity of the ligands has been investigated using DPPH. The fixed reaction time is used to find out antioxidant activity of these three ligands in the solvents.

4.2.3 DPPH Free Radical Scavenging Activity

The antioxidant assay-radical scavenging activity of the ligands has been investigated using DPPH. The fixed reaction time is used to find out antioxidant activity of these three ligands in the solvents. DPPH radical scavenging assay is the most widely used method to evaluate antioxidant activity in relatively short time. Radical scavenging activity of compound A, B and C were evaluated. To determine antioxidant activity solutions of the synthesized compounds in methanol were prepared. Different volumes of this solution is added to definite volume of DPPH in methanol (1M) and made upto 3 ml using methanol. Thus a series of 5 samples were prepared for each compound. The concentration of samples prepared ranges from 1μM-12μM, 10μM-50μM, 6μM-55μM and 2μM-17μM for compound A, B and C respectively. After incubating for 20 minutes, absorbance at 517 nm in measured. Absorbance of control (without adding antioxidant) was also measured. Percentage inhibition is calculated using the following equation

$$\% \text{ inhibition} = (A_{control} - A_{sample} / A_{control}) 100$$

A graph is plotted with concentration against % inhibition. From the plot IC_{50} value (concentration which causes a 50 % inhibition of DPPH free radicals) is calculated. Lower the IC_{50} value greater is the antioxidant activity.

4.3 Results and Discussions

In vitro antioxidant activity of all synthesized ligands were evaluated by DPPH assay, antioxidant activity of the ligands in solvent methanol and corresponding IC_{50} values are also noted. A lower IC_{50} value is indicate of greater antioxidant activity. All the

ligands inhibit DPPH radical in a concentration-dependent manner. The IC_{50} values of ligands in methanol are depicted in Table: 5.

Table: 5 DPPH Scavenging capacities (IC_{50} in µg/ML) of synthesized in methanol solvent.

COMPOUND	A	B	C	BHT
IC_{50}	10.3	14.7	26.2	13

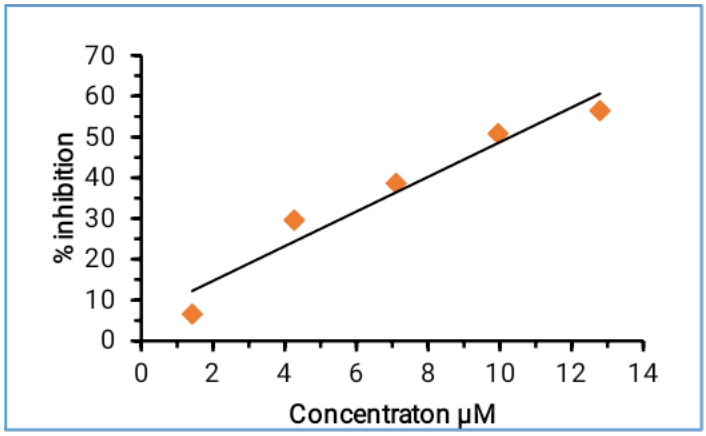


Figure: 24 Compound A; IC_{50}=10.3 µM

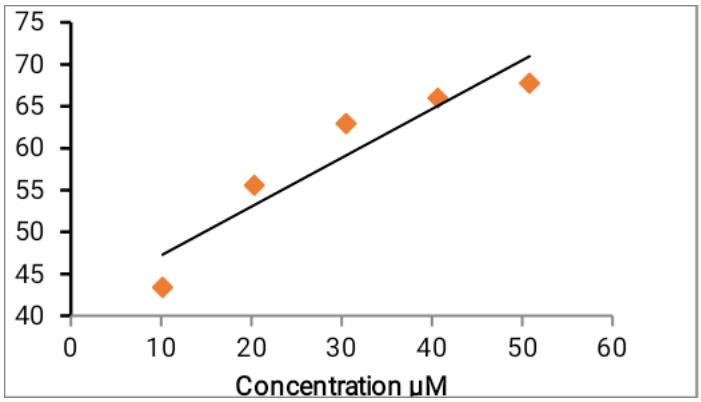


Figure: 25 Compound B; IC_{50}=14.7 µM

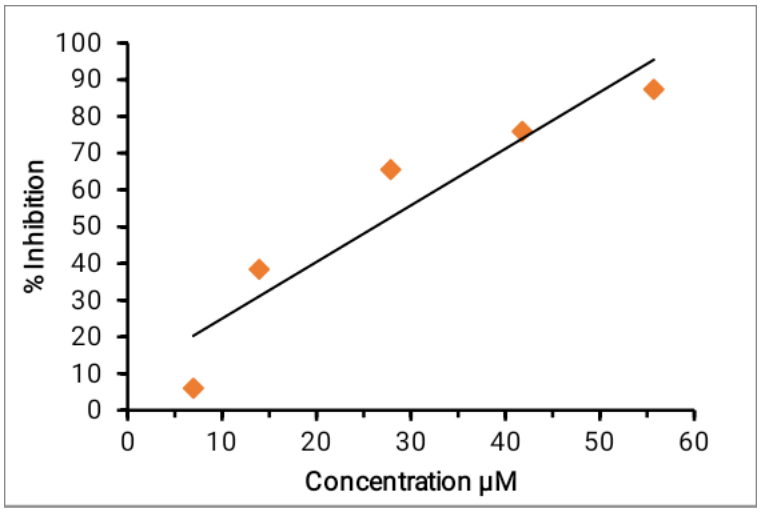


Figure: 26 Compound C; IC_{50}=26.2µM

CONCLUSION

In the present work, the three Novel Schiff bases have been synthesized by the condensation of 2- amino 4- methyl phenol and 4-amino antipyrene with Syringaldehyde and 2,5 dimethyl benzaldehyde are included in the first three chapters. All the synthesized Schiff bases were characterized by IR, UV-VIS, Mass and ^{1}H-NMR spectroscopic methods. All these studies give good evidence for the prepared structure for the Schiff bases. Besides these, this work also evaluates and studied the antioxidant activity of synthesized Schiff bases which is briefly discussed in the last chapter. From the whole it is clear that these synthesized Schiff bases have significant role in diverse biological and pharmacological field.

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