Republic of Iraq
Ministry of Higher Education
And Scientific Research
University of Babylon
College of Science for Women
Department of Chemistry

Synthesis and characterization of new Heterocyclic compounds Based on derivatives of p-Aminobenzoic acid and study some of their Application.

A Thesis

Submitted to the Council of the College of Science for Women,
University of Babylon as a Partial Fulfillment of the
Requirements for the Degree of Master in Science / Chemistry

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2023 A.D. **1444 A.H.**

بسم الله الرحمن الرحيم

(يرفع الله الذين أمنوا منكم والذين أوتوا العلم درجات والله بما تعملون خبير)

المجادلة :(11)

صدق الله العلي العظيم

Supervisor's Certification

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" Synthesis and Characterization of New Heterocyclic Compounds and study their Applications"

was prepared by

" Sura Sadiq ALsultany"

under my supervision at the Department of Chemistry / College of Science for women /University of Babylon as a partial fulfillment of the requirements for the Degree of Master in Science / Chemistry.

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Acknowledgement

Above all else, I want to express my great thanks to ALLAH for the uncountable gifts and for helping me to present this thesis.

I wish to express my sincere gratitude and great appreciation to my Supervisor, Asst Prof. Dr. Nour Abd Alrazzak for her guidance and her encouragement throughout this work.

I would like to thanks the Head department of chemistry Prof. Dr.Sadiq AbdulHussain and Postgraduate cource Dr.Suad Taha and staff of the department of Chemistry.

I would like to thanks my family and all friends for all kind of help they introduced and all the time that they spent with me to finish my work.

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Abstract

This study includes synthesized of new heterocyclic compounds used *p*-aminobenzoic acid and two kind of ketone as starting materials to produce two derivative of chalcone compounds which use to synthesis of new different five , six and seven member heterocyclic compound rings[S3-S9,S12-S18] .Also this study involve synthesis of new different Schiff base heterocyclic compounds [S20-S25] from 1,3,4-thiadiazole amine [S19]. The synthesized organic materials were characterized using different technique: FT-IR, ^{1}H-NMR and ^{13}C-NMR spectroscopy as well as CHNS analysis .

This work is divided into four different parts :

First part:

Synthesis of new azo dye[S1] from reaction between *p*-aminobenzoicacid and 2,4-dihydroxyacetophenone ,then this azo reacted with aromatic aldehyde (3-hydroxy-4-methoxy benzaldehyd) to produce chalcon drevatives [S2]. This chalcon reacted with different compounds to synthesized new heterocyclic compounds ,as shown in scheme (1).

Second part:

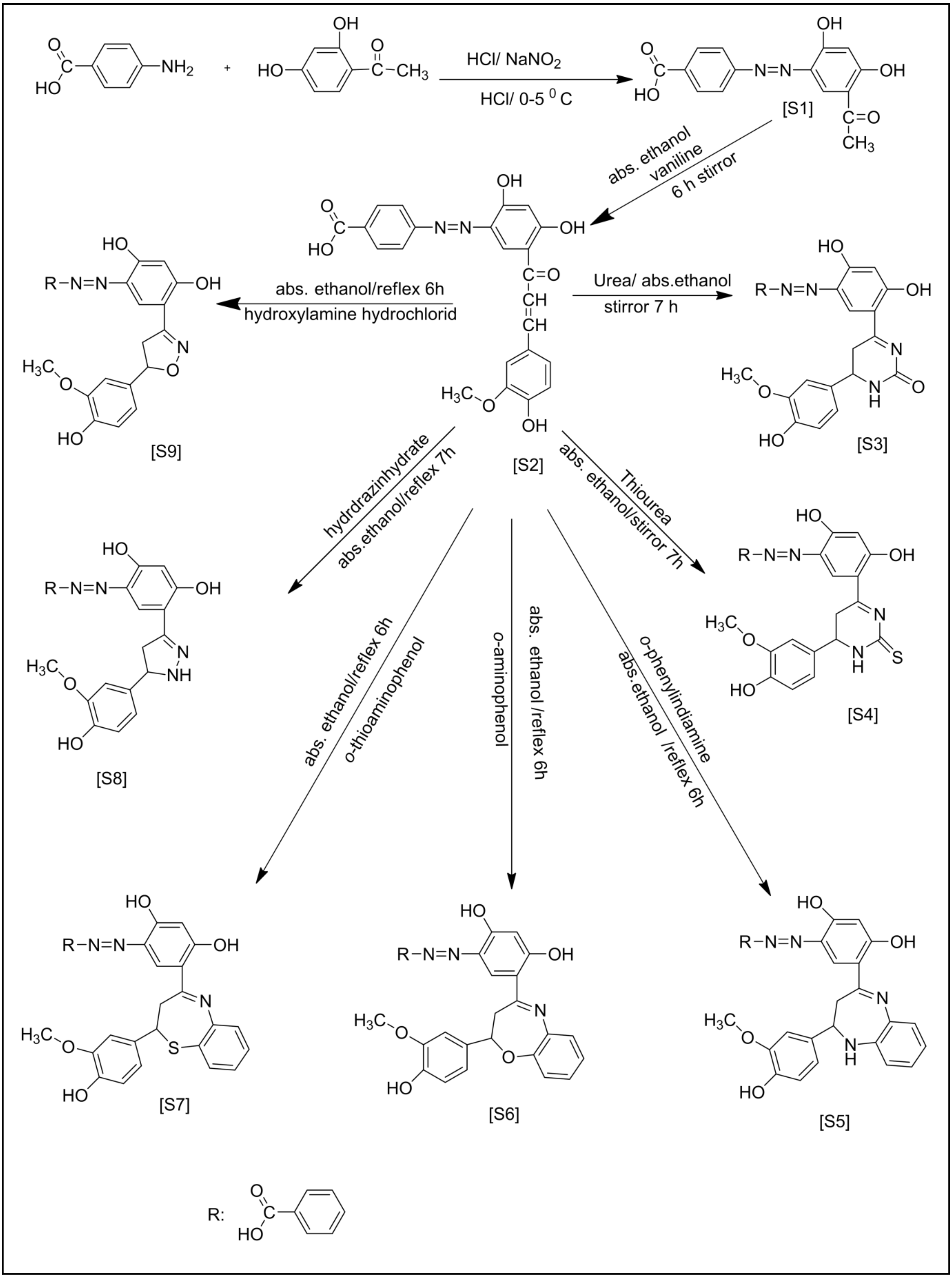
New azo compound[S10] synthesized from interaction between *p*-aminbenzoic acid and 4-hydroxyacetophenone , this azo condensation with aldehyde derivatives(3-hydroxy-4-methoxybenzaldehyd) to produce a new chalcon [S11] Gleason-Schmidt ,then it react with different compounds to synthesized a heterocyclic compounds [S12-S18] scheme (2)show all these reactions.

Third part:

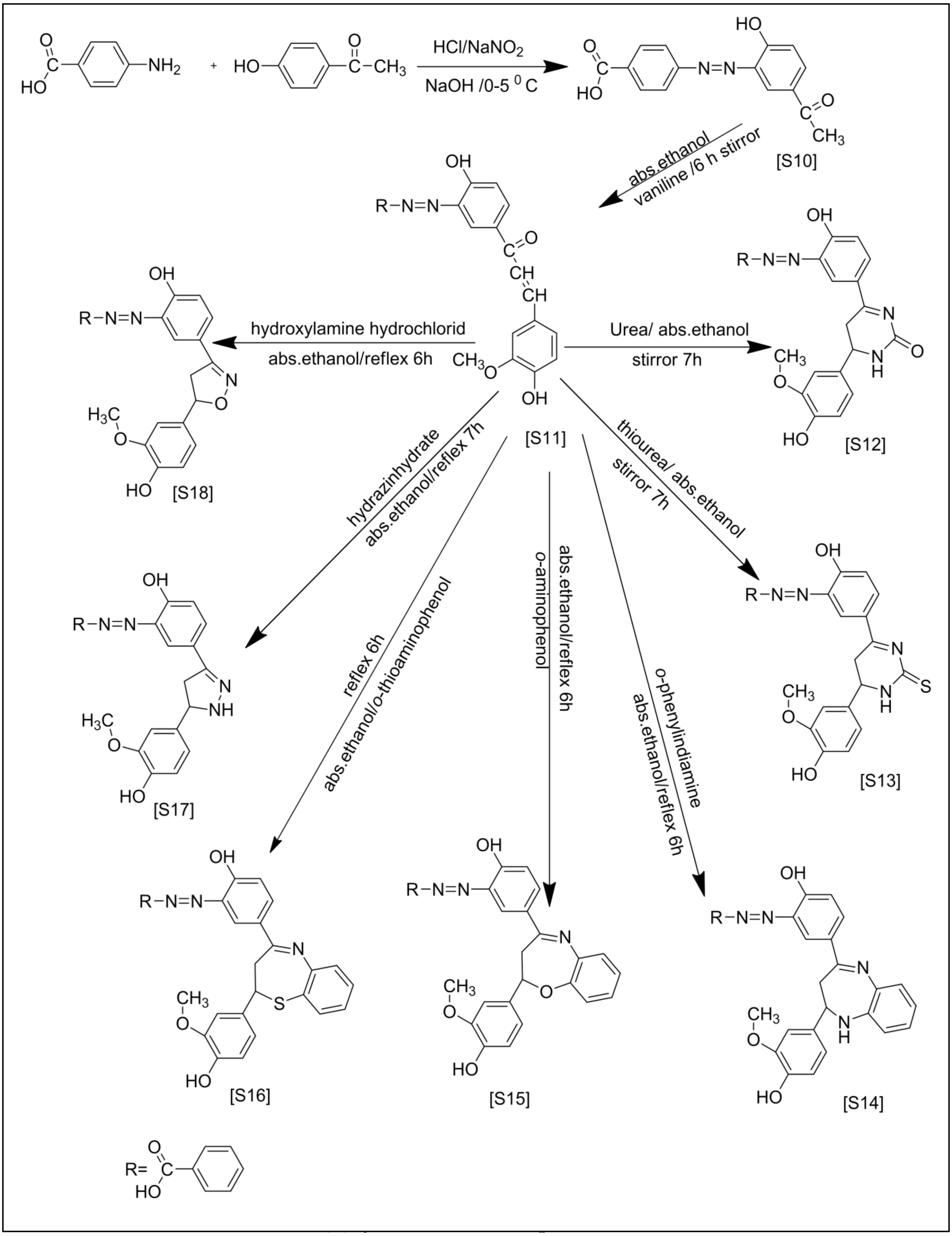
This part involve the synthesized of thiadiazole amine[S19] by reaction between [S10] and thiocemicarbazide in presence of Phosphorusoxychloride .This amine react with different aldehyde to generate new Schiff bases compounds [S20-S21].

Fourth part :

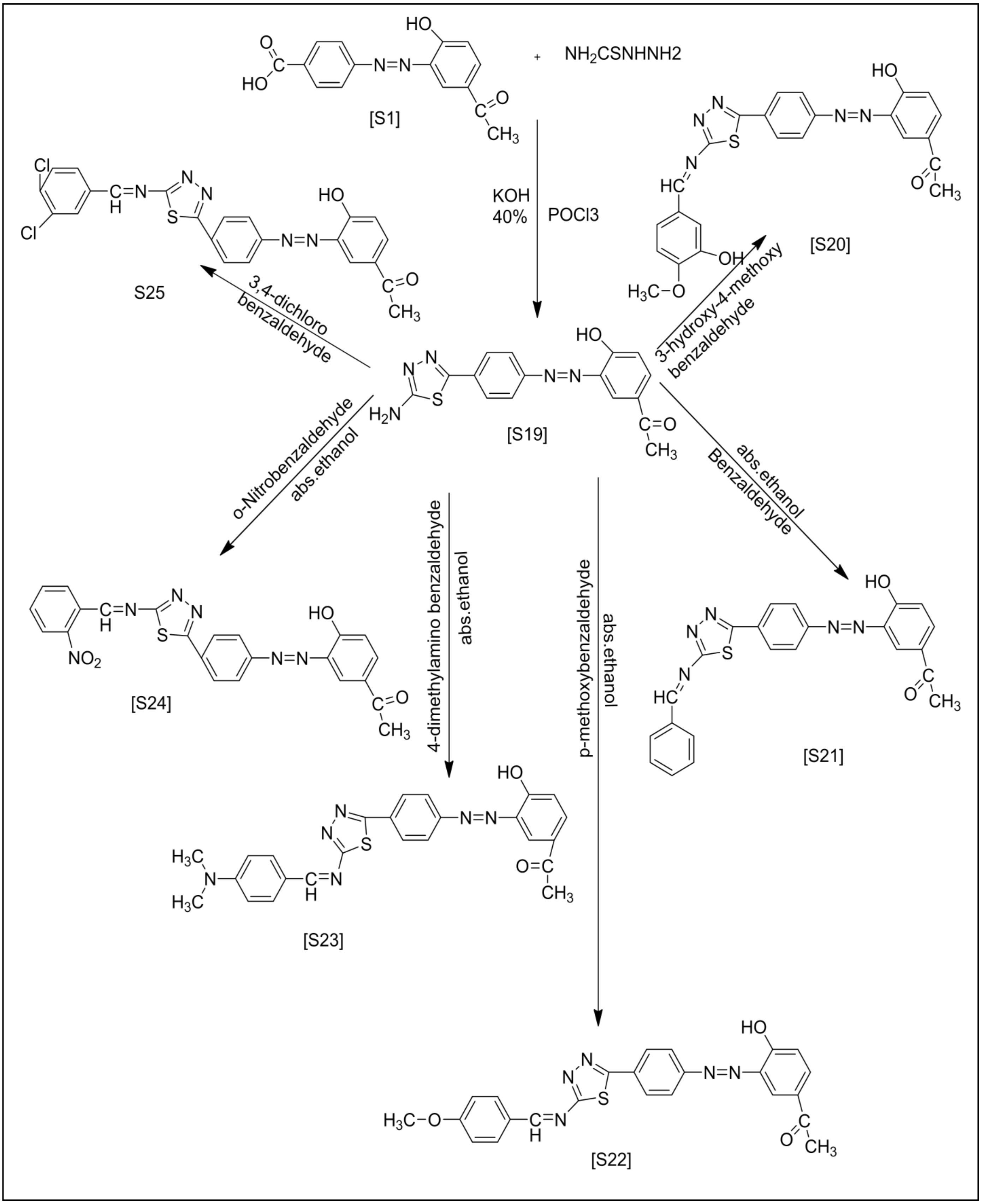
In this part, the biological activity of some types of synthesized compounds was studied against four types of bacteria , two of which are gram positive (*Staphylococcus Epiderimidis, pseudomonas*) and two of which are gram negative (*Klebsiella pneumoniae, and E.Coli.*), as well as an anti-corrosion and anti-cancer study for some of these synthesized compounds.



Scheme (1) synthesis of compounds [S1-S9]



Scheme (2)synthesis of compounds[S10-S18]



Scheme(3) synthesis of compounds [S19-S25]

List of Contents

Chapter Three

Directory of Abbreviations

Symbol	Description
FT-IR	Infrared
^{1}HNMR	Proton nuclear magnetic resonance
^{13}CNMR	Carbon nuclear magnetic resonance
DMSO	Dimethyl sulfoxide
DCM	Dichloro methan
Abs-ETOH	Absolut ethanol
CHNS	Carbon,Hydrogen ,Nitrogen and Sulfur
$POCl_3$	Phophorus oxychloride
$Ga(OTF)_3$	Gadolinium(*III*) trifluoromethanesulfonate
TMS	Tetra methyl Silane
M.F	Molecular formula
M.wt	Molecular wighte
Eocp	Establish steady state open circuit potential
IE	The inhibition efficiency
Icorr	Corrosion current density
Ecorr	Corrosion potential
BZD_S	Benzodiazepines
Et_3N	Triethylamine
MTT	[3-(4,5-dimethylthiazol-2-yl)-2, 5-diphenyltetrazolium Bromide]

List of tables

1. Introduction

1.1. Heterocyclic compounds

Heterocyclic chemistry history began in the 1800, at the same time as organic chemistry. Some notable developments include: Alloxan is isolated from uric acid in 1818, starch and sulfuric acid are combined to make furfural in 1832, and pyrrole (often referred to as "fiery oil") is isolated in 1834 by a furan runge using bone dry distillation. A significant portion of the agriculture business was replaced by synthetic chemistry techniques after Friedlander's discovery of indigo dye in 1906 Treibs explains the biological origin of petroleum in 1936 by synthesizing chlorophyll derivatives from crude oil(1) ·The conventional organic category of chemistry known as heterocyclic is the most significant, and because of its structural Skelton components, they have significant physiological and industrial significance(2)

The term "heterocyclic compounds" refers to cyclic organic compounds that contain at least one heteroatom. The most prevalent heteroatoms are nitrogen, oxygen, and sulfide, but heterocyclic rings with other hetero atoms are also well-known(3).

In nature, heterocycles make up a significant class of chemical substances that are found in alkaloids, hormones, vitamins, and other natural substances(4) Synthetic heterocyclic chemistry has not only played an important role in every place of human life and also found their application in diverse field as agriculture medicine, polymer and various industries(5). More than 90% of the novel drugs bear heterocyclics and among them, nitrogencontaining heterocyclic compounds show superior pharmaceutical effect than non-nitrogen compounds. Nitrogen-containing compounds, the heart of drug discovery(6)

Based on the structural and electronic arrangement the heterocyclic compounds may be classified into two categories:-

i. Aliphatic heterocyclic compounds

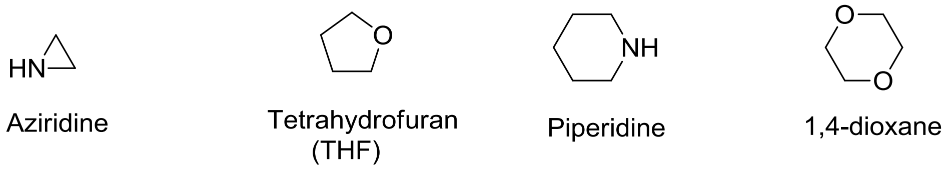


Figure (1-1) aliphatic heterocyclic compounds

ii. Aromatic heterocyclic compounds(7)

Figure (1-2) aromatic heterocyclic compounds

Heterocyclic compounds, particularly those with sulfur and nitrogen atoms, are particularly attractive for research in the planning of organic synthesis due to their biological features(8)

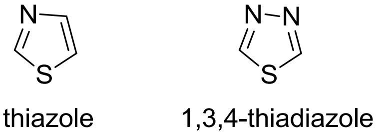
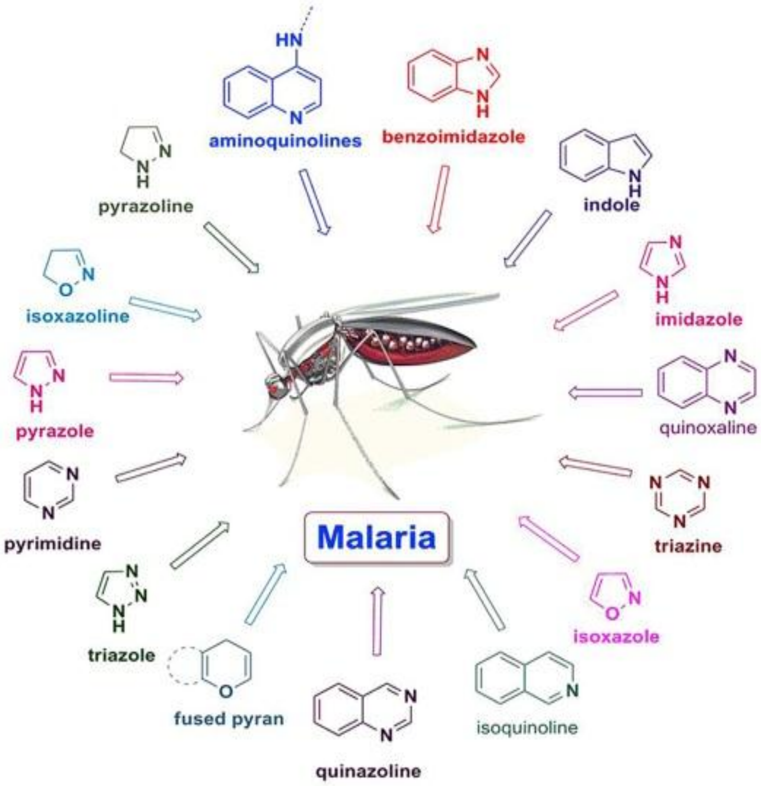


Figure (1-3)

Five-membered nitrogen heterocyclic compounds are regarded to be highly significant structural elements and physiologically active molecules (9-13)

The production of industrial substances including pesticides, corrosion inhibitors, dyes, acid-base indicators, and others depends on them as well(14-18)

Heterocyclic substances are abundant in nature, essential to life, and they play a dynamic part in every living cell's metabolism. DNA is also a collection of heterocyclic bases, such as pyrimidines and purines(19)and it concederd as anti malarial agent , figure (1-4) show virous heterocyclic compound that involve some malarial drugs(20)



Figure(1-4) some of malarial drugs

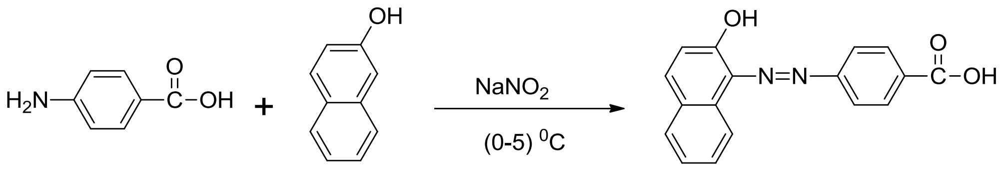
1.2. Azo Dye Compounds:-

Azo dyes, which are distinguished by the presence of (-N=N-) in their structural formula, are one of the most important groups of organic compounds (21-22) In addition to auxochromic groups like (OH, NH_2, NHR, and NR_2) that are present as substituents on the aromatic rings in the azo compound structure, azo compounds also contain a chromophoric group (azo group -N=N-). Because of their vibrant hue, azo dyes are employed as paints in the textile and plastic industries (23).

Azo compounds are adaptable substances utilized in many different applications, including batteries and anti-cancer medications. For instance, Ganguly and et al, observed that the copper complex of an azo molecule demonstrated antitumor action by tightly binding to DNA(24) Additionally, they have been used in a variety of industries, including liquid crystal displays, cosmetics, food coloring, plastics, optical switches, optical data storage, nonlinear optics, and electro-optical devices(25).

1.2.1. Synthesis of Azo Compounds :

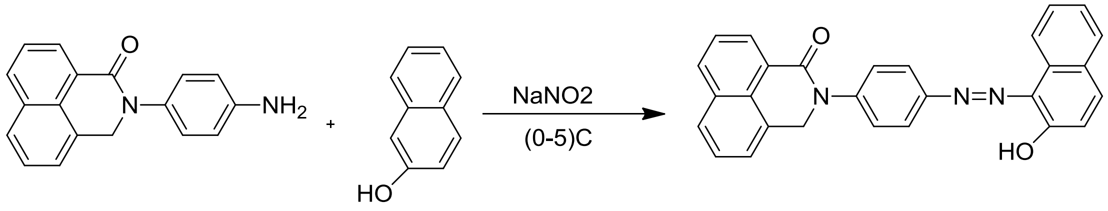
N.A. Alrazzaq and *et. al*. were prepared compound [3] from reaction *p*-amino benzoic acid [1] with 2-naphthol [2]at (0-5 ^{0}C) (26)



[1] [2] [3]

Equation (1-1) synthesis of compound[3]

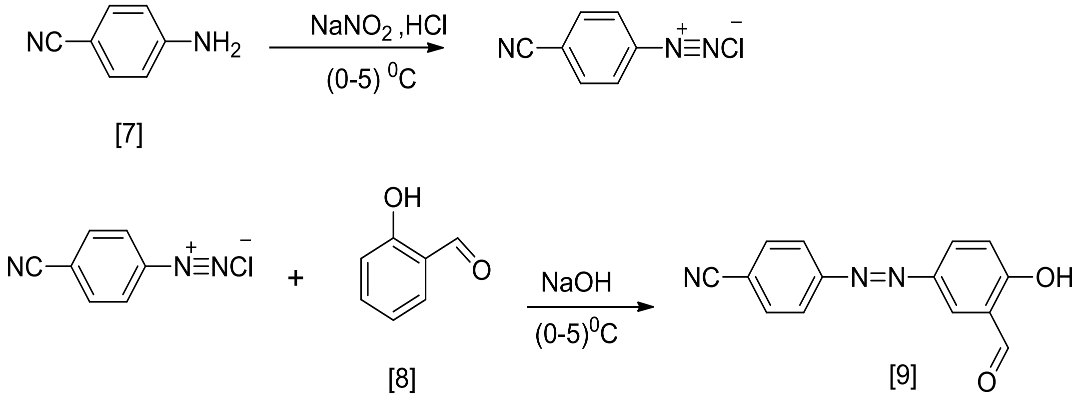
M. G. and *et* al were prepared azo compounds from reaction different subsitituted amine[4] with 2-Naphthol [5] in presence of acetic acid and sodium nitrite at(0-5) ^{0} C (27)



[4] [5] [6]

Equation (1-2) synthesis of compound[6]

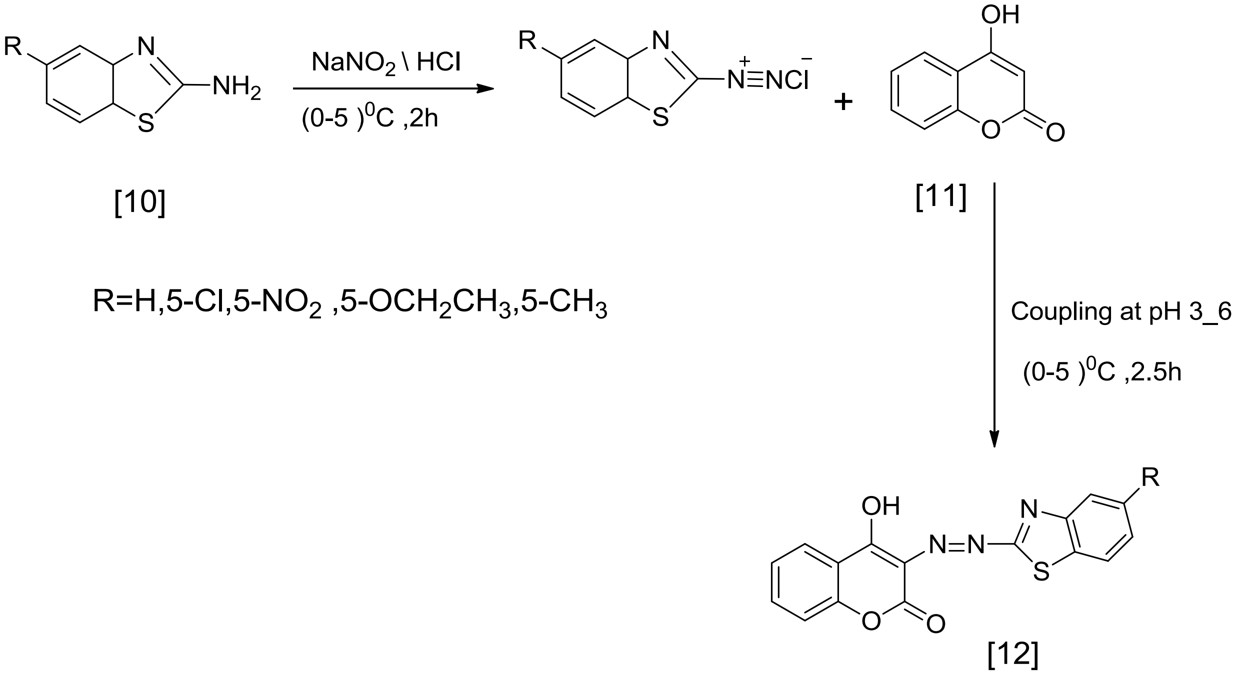
A.A. and *et.al*. were prepared compound from reaction 4-aminobenzonitrile with salicyladehyde at (0-5) ^{0}C(28) .



[7] [8] [9]

Scheme (1-3) synthesis of compound[9]

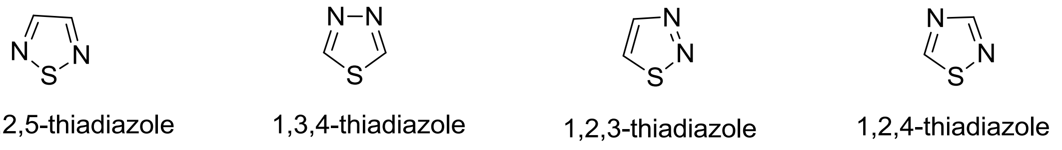
Manjunatha and *et.al.* were prepared derivatives of compound [12] by reaction of 2-amino substituted benzothia-zole [10] with 4-hydroxy coumarin [11] at (0-5)^{0}C and in presence HCl,$NaNO_2$ then KOH $^{(29)}$.



Scheme(1-4) synthesis of compound[12]

1.3. 1,3,4-Thiadiazole:

Thiadiazoles have an unsaturated ring-shaped structure made up of two carbon, three nitrogen, and one sulfur atoms$^{(30)}$.Five-membered heterocyclic compounds called thiadiazoles are quite rare in nature. Numerous isomers, including 1,2,3, 1,2,4, 1,2,5, and 1,3,4-thiadiazole, may be produced by positioning nitrogen, sulfur, and carbon atoms in such a ring in different ways$^{(31)}$.



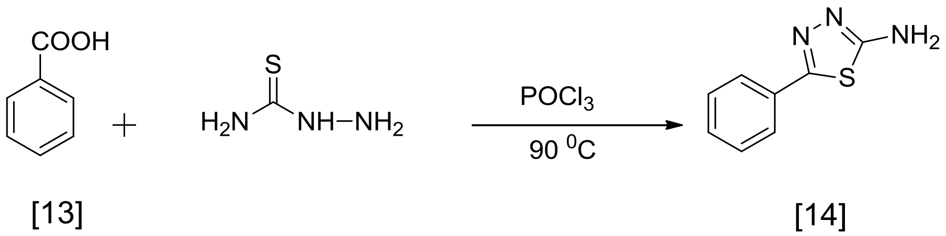
1,2,5-thiadiazole 1,3,4-thiadiazole 1,2,3-thiadiazole 1,2,4-thiadiazole

Figure (1-5) isomers of thiadiazole

Literature supports indicates that thiadiazole and their derivatives have been board range biological actions including antifungal ,antimicrobial, anti-inflammatory ,anti-tubercular, anti-convulsant , anti-cancer, anti-diabetics and antiviral activities .Many drugs containing 1, 3, 4-thiadiazole moiety are commercial available in medicine stores such as acetazolamide(32-38).

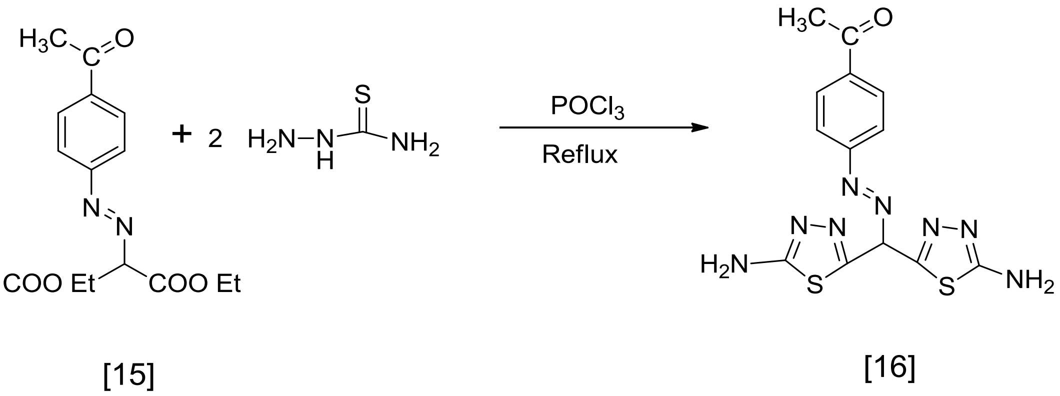
1.3.1 Synthesis of 1,3,4-Thiadiazole Compounds :

1,3,4-thiadiazole[14] was created by Chinnagiri and et al. by reacting benzoic acid [13] with thiosemicarbazide in the presence of $POCl_3$ reflux for 8 hours(39).



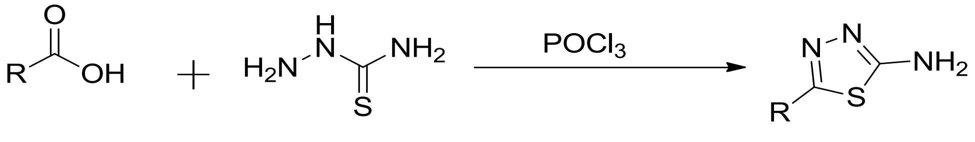
Equation (1-5) synthesis of compound[14]

1,3,4-thiadiazole was created by N.M. and colleagues by reacting an azo chemical with thiosemicarbazide while using $POCl_3$ and reflux for seven hours(40).



Equation (1-6) synthesis of compound[16]

1,3,4-Thaidiazole was created by Tatiana and et al by reacting carboxylic acid with thiosemicarbazide in the presence of $POCl_3$ and reflux for 10 hours$^{(41)}$.

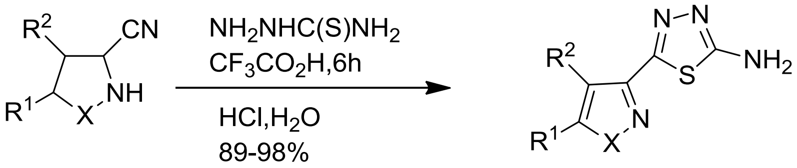


[17] [18]

R= benzoic acid ,3-phenylpropionic acid ,phenoxyacetic acid ,2-Naphthaleneacetic acid , adipic acid

Equation (1-7) synthesis of compound[18]

Sergey K. Petkevich1and *et al* obtain 5-azolyl-1,3,4-thiadiazol-2-amines [20] by used an alternative synthesis method involving the reaction of acid nitriles with thiosemicarbazide in CF_3CO_2H. The target aminothiadiazoles were obtained in 89–98% yields$^{(42)}$.



[19] [20]

R^1=Ph,4-MeC_6H_4,Cl
R^2=H,Cl
X=O,S

Equation (1-8) synthesis of compound[20]

1.4.The Chalcones

1,3-Diphenyl-1-propen-1-one, often known as chalcone ($C_{15}H_{12}O$), is a crucial component of nature. Chalcones have one unsaturated carbon atom and two aromatic rings (A and B) and one carbon atom α, β-unsaturated$^{(43)}$.

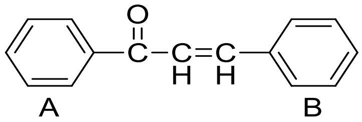


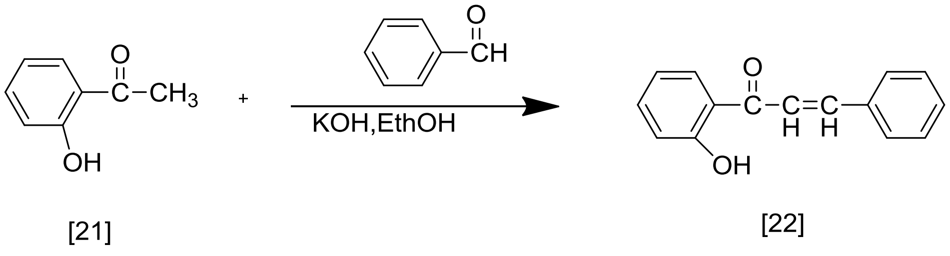
Figure (6) structure of chalcone

As a result, chalcones are phenyl-styryl ketones with a reactive ketoethylenic group -C-COCH- and are distinguished from other types of ketones by their location in the Ar(A)-CO-CH- CH-Ar(B) structure(44) Because of their wide pharmacological possibilities, including actions and features, natural and synthesized chalcones have received a lot of attention in medicinal chemistry research in the 21st century(45) for instance, antibiotics(46-47) anti-inflammatory(48) antioxidant(49), anti-platelet(50), anti-cholinergic(51), and anti-inflammatory medicines that treat diabetes(52), cancer(53), viruses(54), and leishmaniasis(55).

1.4.1. Synthesis of chalcone Compounds :

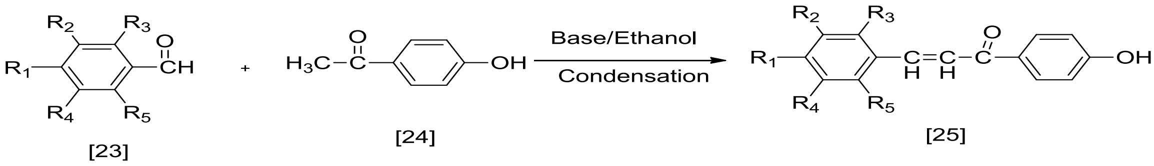
The synthesis of chalcones has been reported using a number of techniques. The most common ones are Claisen-Schmidt condensation and Aldol condensation. Additionally, they are produced using the Suzuki process, Friedel-Crafts acylation, and Wittig reaction(56).

Chetana B. Patil *et al.* have disclosed an unique Aldol type condensation for the synthesis of chalcones(57)



Equation (1-9) synthesis of compound[22]

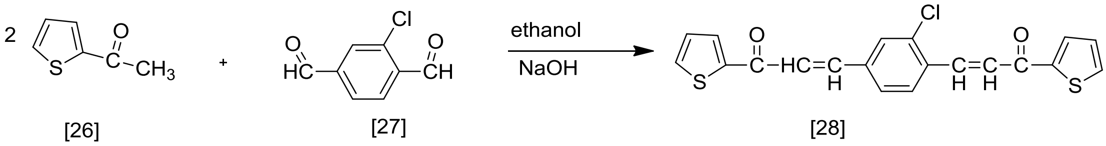
Chalcones are often made from aldehydes and ketones derivatives inorganic base such as sodium hydroxide, potassium using an hydroxide, or alkali alcoholate as a catalyst for aldol condensation(58).



$R_1=NO_2$, OH
$R_2=OCH_3$, OH
$R_3=CH_3$
$R_4=OH$
$R_5=CHO$

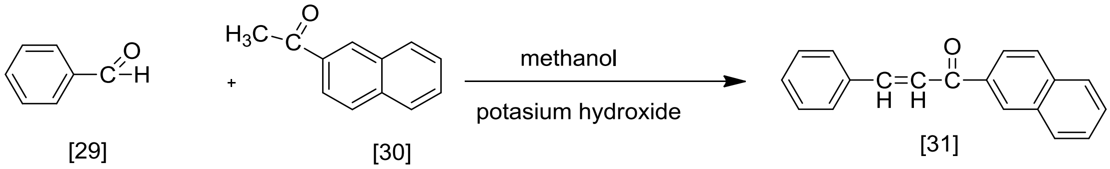
Equation (1-10) synthesis of compound[25]

Al-Rammahi, F. and *e*t al are synthesis chalcone from reaction between 1-(thiophen-2-yl)ethanon[26] and 2-chloroterephthalaldehyde[27] in precense of absolut ethanol and sodium hydroxide(59)



Equation (1-11) synthesis of compound[28]

Nadia A. A. Elkanzi *et al* are synthesis chalcone by treating 2-acetyl naphthalene[30] with benzaldehyde and/or substituted benzaldehyde [29] in methanol and potassium hydroxide.(45)



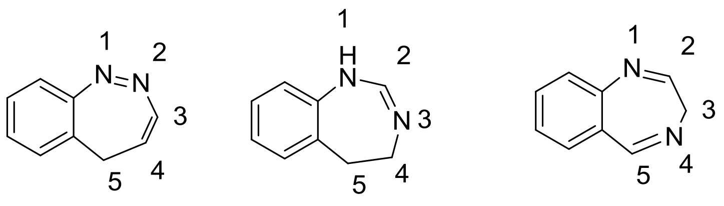
Equation (1-12) synthesis of compound[31]

1.5. Benzodiazepines:

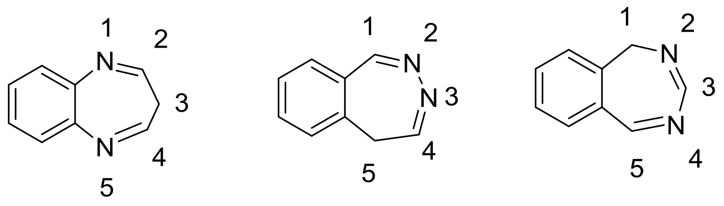
Benzene ring system fused to a se Benzodiazepines are nitrogen heterocycles having a six-membered seven-membered fused ring system with two nitrogen atoms. Because of their improved pharmacological, clinical, and biological features, chalcone-derived benzodiazepine scaffolds remain the main focus by the scientific community(60).

Benzoheterodiazepine is commonly synthesized from chalcone as a precursor via aza-Michael addition reaction into the various derivatives such as benzodiazepine, benzothiazepine, and others(61).

There are various types of BZDs, namely, 1,2-BZDs, 1,3-BZDs, 1,4-BZDs, 1,5-BZDs, 2,3-BZDs, and 2,4-BZDs, but widely used BZDs are 1,4- and 1,5-BZDs. In the nomenclature of BZDs,the nitrogen ring closest to the benzene ring is given priority,so the numbering of various BZDs in the simplest form is given in figure(11)



5H-1,2-BZD$_S$ 1H-1,3-BZD$_S$ 1H-1,4-BDZ$_S$



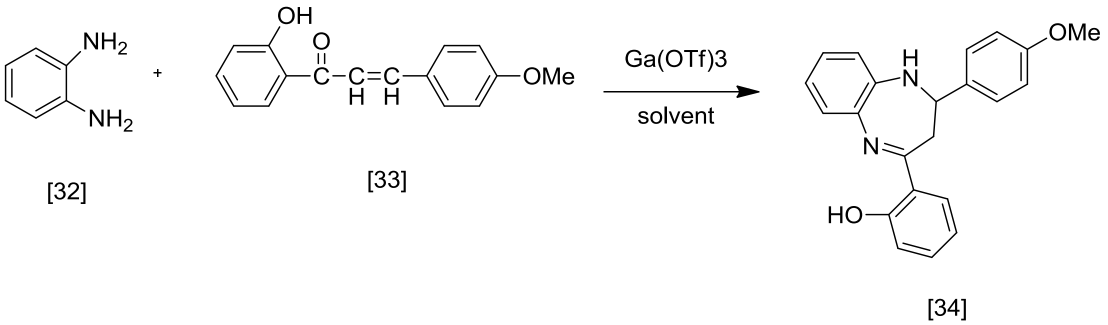
3H-1,5-BZD$_S$ 5H-2,3-BZD$_S$ 1H-2,4-BZD$_S$

Figure (1-7) isomers of BZD$_S$

1,5-BZDs have a wide range of beneficial properties, including amnesia, anticonvulsant, hypnotic, analgesic, antimalarial, antifungal, antibacterial, , antiviral, anti-inflammatory, and more. Anticancer,phytotoxic, and other properties(62).

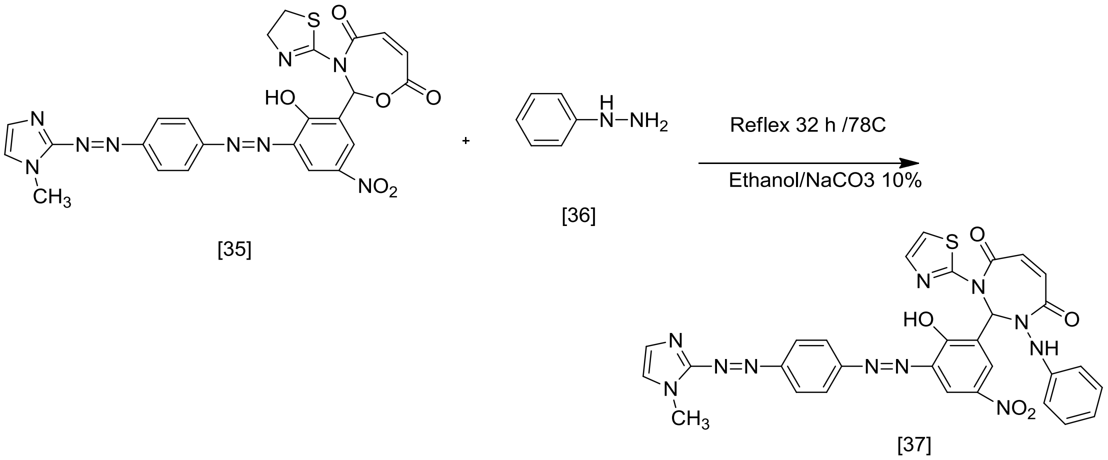
1.5.1 Synthesis of diazepine Compounds :

Pan, X.**,** *et al* synthesis of diazepine compound [34] from treating chalcone compound[33] with *o*-phenylindiamine [32] in precense of Ga(OTF)$_3$ as cateleste(63)



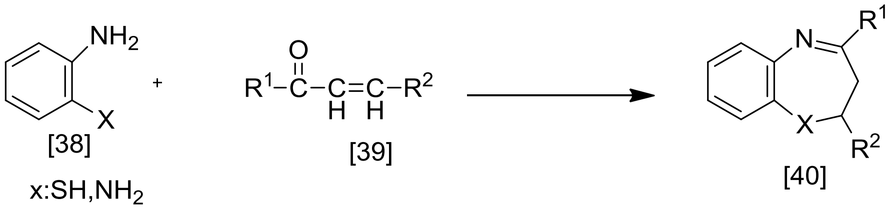
Equation(1-13) synthesis of compound[34]

Mohamed Jawad *et al* are produce diazepine drevitaves[37] by reaction Azo drevitaves compound [35] with phenyl hydrazine compound [36] with reflex for (32 h)at(78c^{0})(64)



Equation(1-14) synthesis of compound[37]

Saba Farooq *et al* synthesis Benzoheterodiazepine [40] from chalcone as precursor via aza-Michael addition reaction into the various derivatives such as benzodiazepine, benzothiazepine, and others. These compounds are commonly synthesized following two-pot and one-pot synthetic routes(61).

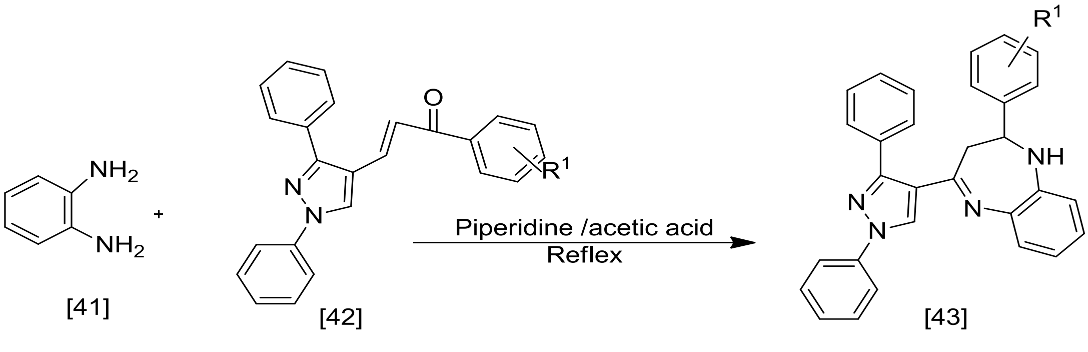


x:SH,NH_2

R^1,R^2:Aryl,Hetro aryl

Equation(1-15) synthesis of compound[40]

Sunita and *et al* synthesis benzodiazepine drevitves [43] by reaction between unsaturated ketone [42] and phenyline diamine[41] in the presence of piperiden and acetic acid with reflex (62)



Equation(1-16) synthesis of compound[43]

1.6. Pyrazol Compounds:

The five-membered ring shape of the unsaturated heterocyclic organic molecule is made up of three carbon elements and two nitrogen atoms. Pyrazole(8)

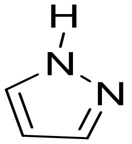
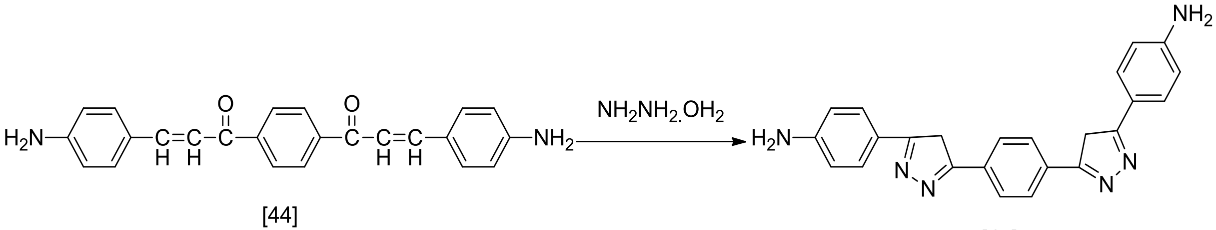


Figure (1-8) structure of pyrazol

There is recognition for the pyrazoles family of heterocyclic compounds for their importance as drug targets and medicinal interest. Biological actions carried out by organic substances with extra S and/or N atoms include antioxidant and antibacterial characteristics(65-69).
Due to its medicinal significance, pyrazolole core is crucial in the pharmaceutical industry for the development of medicines like those that are anticancer, antibacterial, and antifungal(70-73).

1.6.1 Synthesis of Pyrazole Compounds:

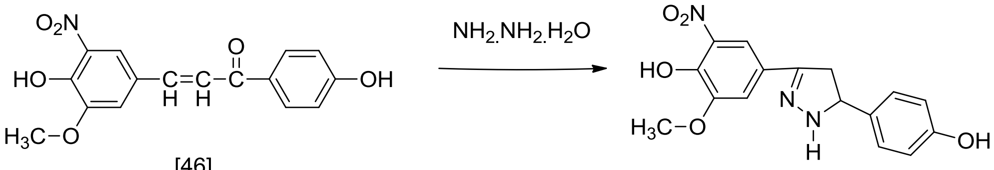
Alaa Jawad K. *et al* Were prepared the compound [45] by reaction the chalcone derivatives [44] with hydrazin hydrate and reflux for 6 hours(74).



[44] [45]

Equation (1-17) synthesis of compound[45]

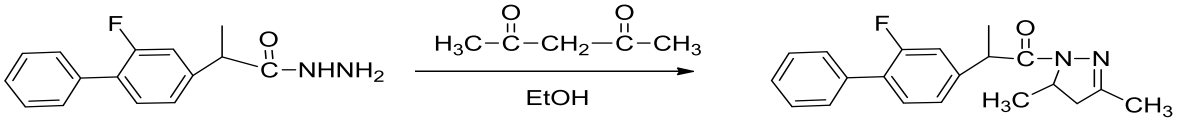
S. A.was prepared the pyrazol [47] by reaction the chalcone [46] with hydrazine hydrate and glacial acetic acid in absolute ethanol and reflux for 15 hours(75).



[46] [47]

Equation (1-18) synthesis of compound[47]

M.Amir and Sh.Kumar were prepared the compound [49] by reaction the acid hydrazide [48] with acetyl aceton in absolute ethanol and reflux for 8 hours(76).



[48] [49]

Equation (1-19) synthesis of compound[49]

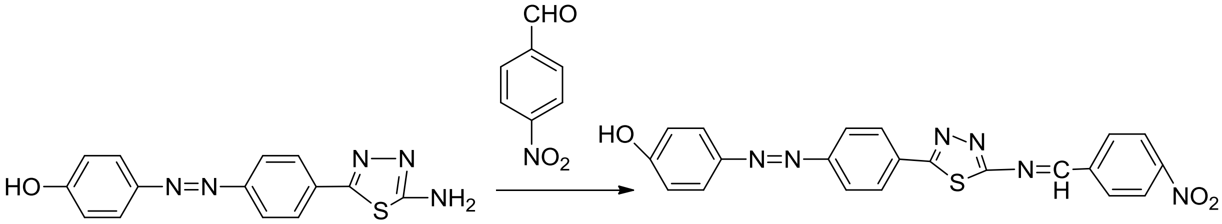
1.7. Schiff Bases:

Due to their simplicity in synthesis, variety of color-rich structures, and interest they have generated in the field of thermochromic materials, schiff bases are now the subject of substantial research(77). Schiff bases are condensation products of primary amines and carbonyl compounds in which the carbonyl group is replaced by an imine or an azomethine (C=N) group. $R_1HC=NR_2$ is a formula that often describes them, where R_1 and R_2 are alkyl or aryl groups(78-81).

In many diverse fields, such as analytical, biological, and inorganic chemistry, the use of Schiff bases has increased. Schiff bases are one of the most commonly used types of organic compounds. Because they have a variety of biologically similar functions and are anti-inflammatory(82)

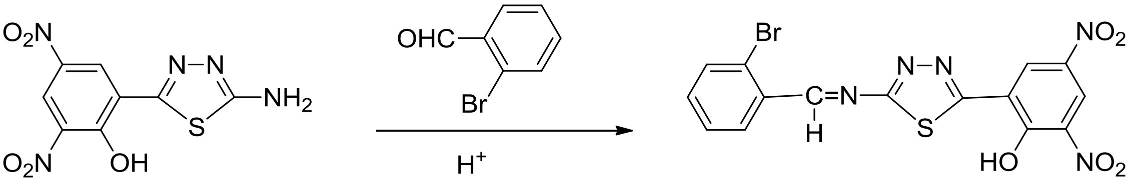
1.7.1 Synthesis of Schiff Base Compounds :

Schiff base [51] was created by Shimaa et al. by reacting 1,3,4-thiadiazole[50] with an aldehyde derivative and refluxing for two to three hours(39).


[50] [51]

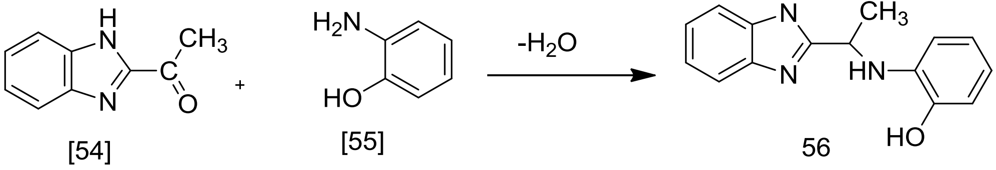
Equation(1-20) synthesis of compound[51]

By reacting 1,3,4-thiadiazole [52] with an aromatic aldehyde in the presence of glacial acetic acid and reflux for six hours, R. Kais and S. Adnan created the shciff base [53](99).


[52] [53]

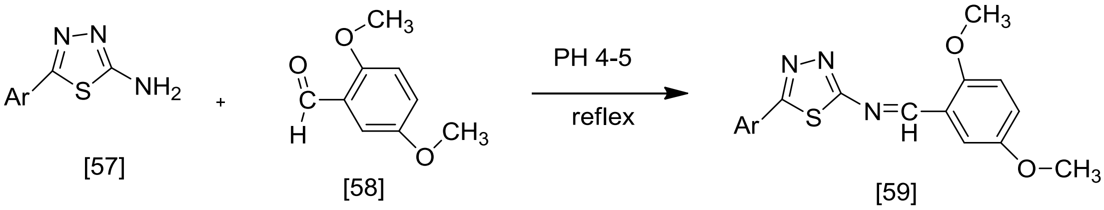
Equation(1-21) synthesis of compound[53]

N-(methyl-2-benzimidazolmethylidene)-2-hydroxyanaline [56],a new Schiff base synthesized by Hu et al. via the condensation reaction of 2-acetylbenzimidazole [54] and 2-aminophenol [55](84).


[54] [55] 56

Equation(1-22) synthesis of compound[56]

Mahmut and *et-al* were created Schiff base [59] using a pair of (2,3-dimethoxybenzaldehyde [58] and 2-amino-1,3,4-thiadiazoles [57] (85).



Equation(1-23) synthesis of compound[59]

1.8. Pyrimidine compounds:

Pyrimidine is a crucial six-membered compound found in heterocyclic chemistry and a member of the alkaloids family. It plays a crucial part in a number of biological processes purine, cytosine, uracil, thymine, folic acid, and coenzymes are all processed as vitamins(61).

Pyrimidines are heterocyclic substances with a ring structure made up of two nitrogen atoms and four carbon atoms. Furthermore, it takes the shape of 3,1-heterocyclic hexagonal diazines(86).

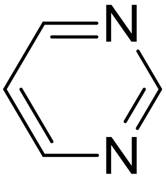
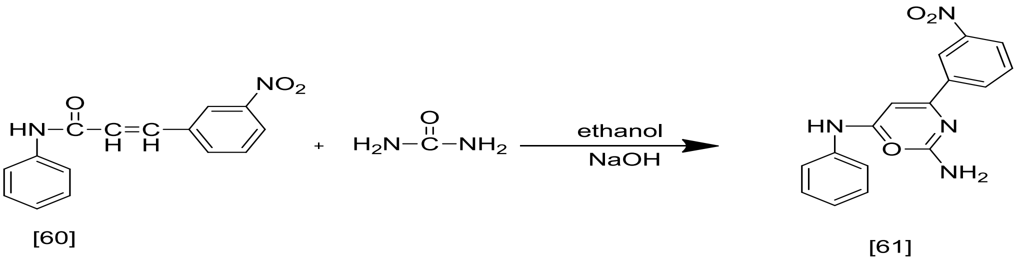


Figure (1-9) structure of Pyrimidine

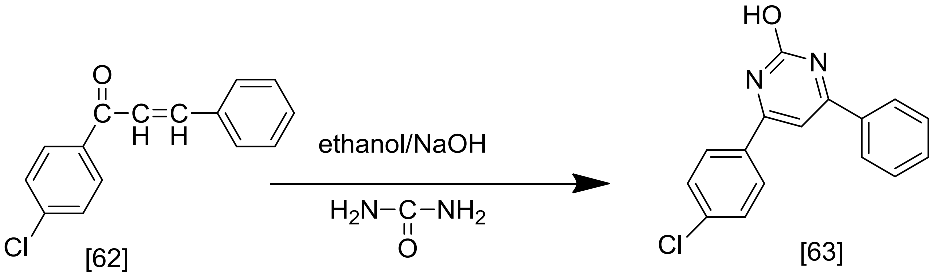
1.8.1. Synthesis of Pyrimidines compounds:

Zainab Sallal synthesis Pyrimidine compound [61] by reaction between chalcone [60] and urea in precense of sodium hydroxide and ethanol(87)



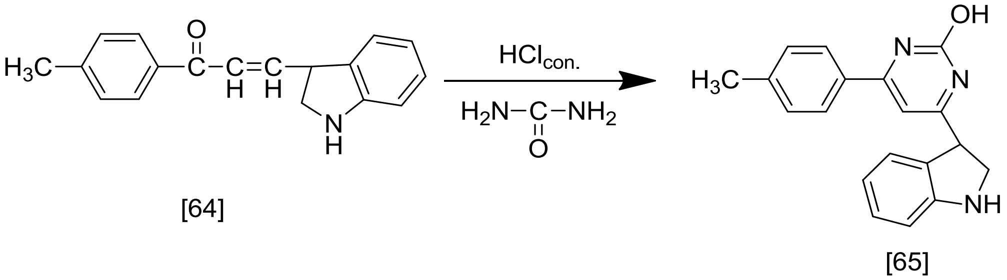
Equation(1-24) synthesis of compound[61]

Rana *et al* synthesis Pyrimidine [63]by reaction the chalcone [62]derivative with urea and with stirror for (2-3 h)(88)



Equation(1-25) synthesis of compound[63]

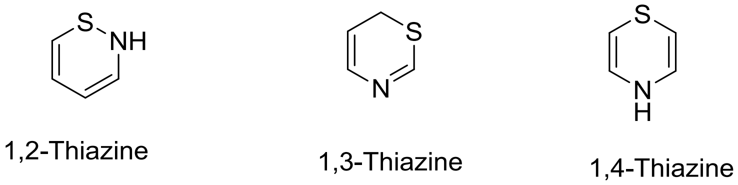
Panda SS *et al* synthesis of Pyrimidine[65] when the reaction between chalcon and urea has accure in precense of concentration hydrochloride acid(89).



Equation(1-26) synthesis of compound[65]

1.9.Thiazine Compounds:

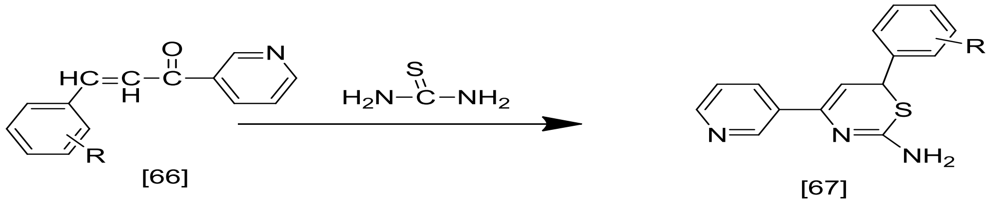
A thiazine compound has a ring of four carbon atoms, one nitrogen atom, and one sulfur atom. Thiazine has three isomers: 1,2-thiazine, 1,3-thiazine, and 1,4-thiazine. These three are distinct from one another by the placement of the nitrogen and sulfur atoms in the ring he N-C-S linkage in the structure of 1, 3-thiazines is acknowledged to be a particularly valuable unit in the disciplines of medical and pharmaceutical chemistry. There have been reports of a range of biological activity for 1,3-thiazines and its derivatives, including antibacterial and antifungal effects. **(90).**



Figure(1-10) isomers of thiazine

1.9.1.Synthesis of thiazine Compounds:

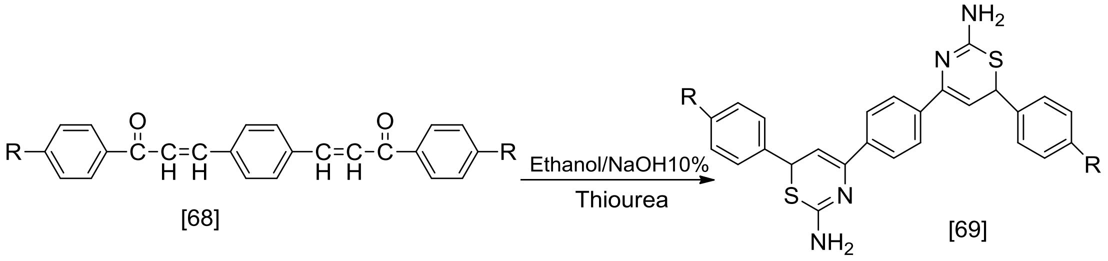
Syed *et al* synthesis the thiazine compound [67] by reaction between azachalcone [66] with thiourea(91)



R=*m*-NO_2,*P*-OH,*m*-Br

Equation(1-27) synthesis of compound[67]

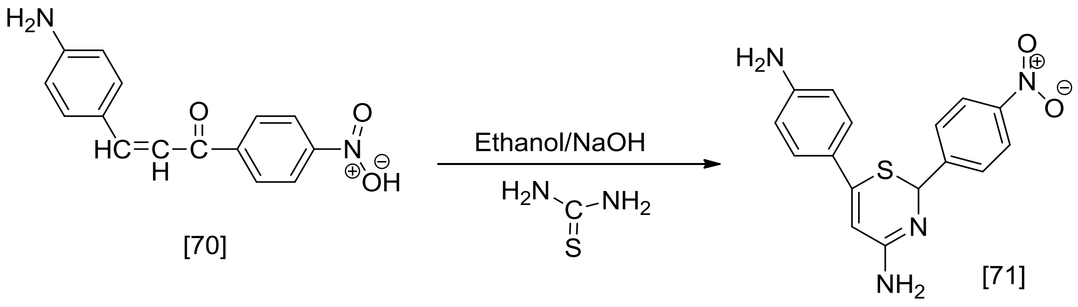
Ameen Synthesis thiazine derivative[69] by reaction chalcone [68] with thiourea and ethanol ,10% sodiumhydroxide as catalest.(92)



R=OH,OCH_3,Br,Cl

Equation(1-28) synthesis of compound[69]

Hala M. Sabre,created thiazine compound [71] when chalcone [70] and thiourea had react in presence of ethanol and sodium hydroxide(93)



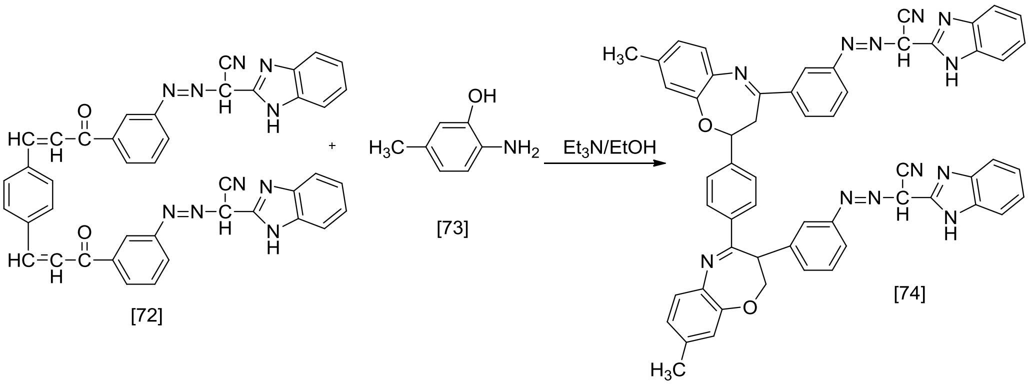
Equation(1-29) synthesis of compound[71]

1.10.Oxazipen Compounds:

A seven-membered ring called an oxyazepine comprises five carbon atoms as well as oxygen and nitrogen. There are many variants of the heterocyclic oxazepine. Over time, studies and reports on oxazepine synthesis have been made**(94)** . Heterogeneous organic compounds with nitrogen in their composition have a wide range of medicinal uses. Oxazepine derivatives are one among them. Oxazepine molecules exist in three isomers: (1, 2), (1, 3), and (1, 4). This numbering depends on the location of the oxygen andnitrogen atoms in the seven-membered ring , When compared to the benzene ring, the ring is asymmetrical due to the increase in size.
In order to alleviate the stress on the ring and make it more stable, the distribution of vacuum atoms causes it to adopt the shape of a boat(95).

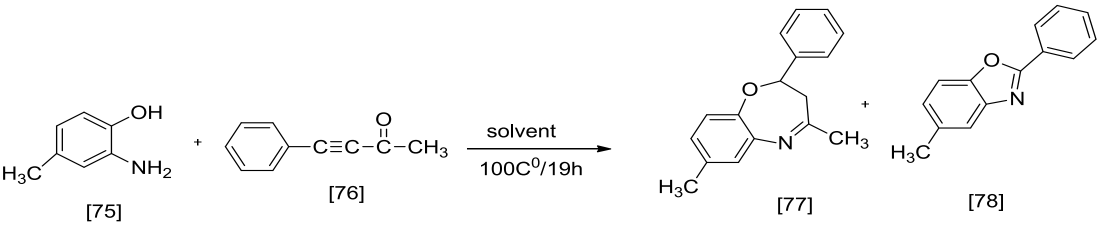
1.10.1. Synthesis of Oxazipen Compounds:

Fatin *et al* synthesis oxazipen derivative[74] by reaction of chalcone derivative [72] with ortho amino phenol [73] in presence of trimethylamine and ethanol(96).



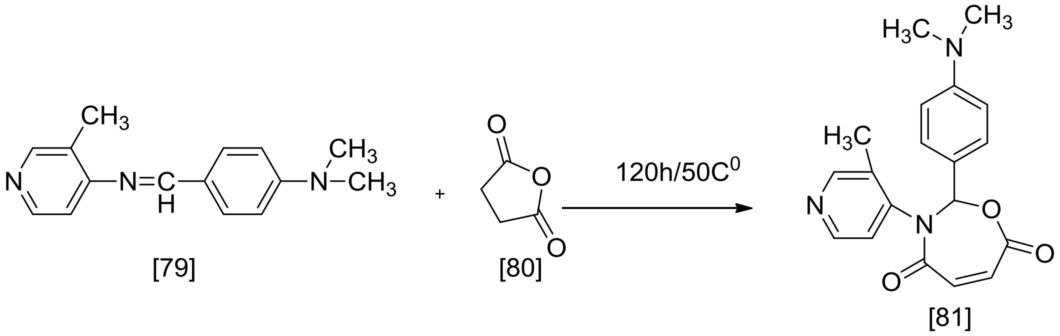
Equation(1-30) synthesis of compound[74]

Kohei etal synthesis oxazipen compounds [77] by reaction of 4-methyl-2-aminophenol [75] with 1.2 equivalents of alkynone [76] in reflex for 19 h at 100C^{0}(97).



Equation(1-31) synthesis of compound[78]

Mohaned M.*et al* synthesis oxazipen compound [81]by reaction of Schiff base [79] and compound [80] with reflex for 120h at 50C(96)



Equation(1-32) synthesis of compound[81]

1.11. The biological activity:

Azo dyes have special biological functions as cleaning agents, chemotherapeutics, anti-inflammatory, anti-cancer, and anti-diabetic medications. Azo compounds are extremely colorful and have been used as dyes for pigment for a very long time, but due to their environmental resilience, electrical properties, and optical properties, azo compounds are of remarkable relevance(97-99).

Azo dye compounds are used in a variety of industrial processes, such as photodynamic therapy, and have biological activity due to their employment in inflammatory, anticancer, antibacterial, and antifungal activities(100).

Recent reviews have examined the effects of pyridazine derivatives on analgesics and inflammation, antibiotics, epilepsy, cardiovascular disorders, and cancer(101-105). Because of its therapeutic value, pyrazole core is crucial in the pharmaceutical industry for the creation of medications that are anticancer, antibacterial, antimicrobial, antifungal, etc (106-108) . It has been demonstrated that Schiff bases serve a number of biological purposes. These biological activities include those that have antifungal, antibacterial, antimalarial, anti-proliferative, anti-inflammatory, antiviral, and antipyretic properties(38).

1.12 Corrosion and it's Effect:

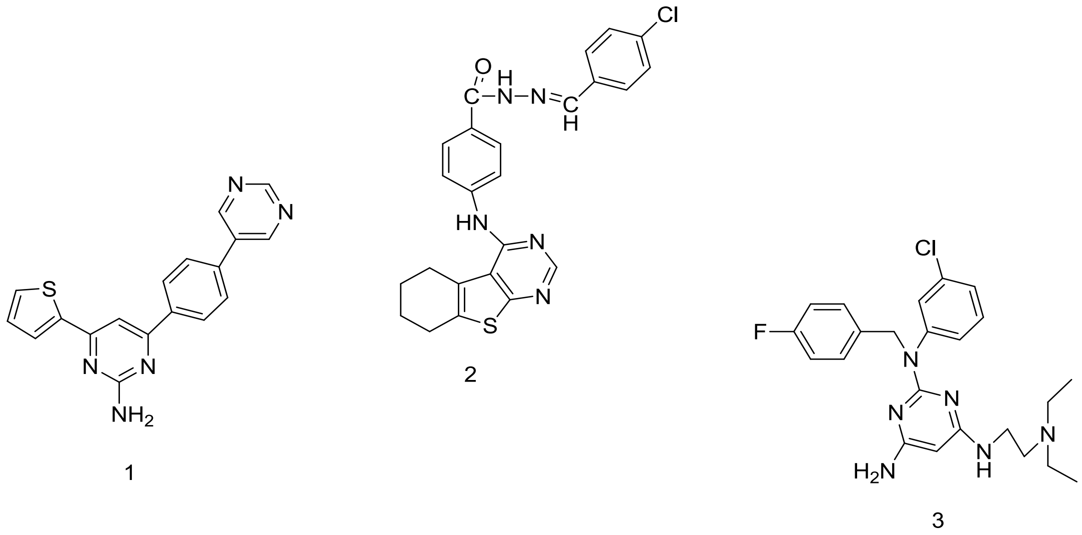
Corrosion is the deterioration that metals and alloys sustain as a result of chemical or electrochemical interaction with their surroundings(109).

According to the different types of corrosive environments, corrosion reactions are divided into two categories: wet and dry corrosion(110-111).

Corrosion is an issue that affects all uses of metals. The cost of maintaining and safeguarding the utilised materials increases due to corrosion damage. The creation of methods to control corrosion presents a challenge for researchers in this area. One of the attractive and most effective corrosion control methods for metals in contact with corrosive environments is the use of an inhibitor. Inhibitors reduce the corrosion of metallic materials by restricting the dissolution and consumption of metal. The bulk of the well-known inhibitors for the corrosion of steel in acidic conditions are organic chemicals containing nitrogen, sulphur, oxygen, or N-heterocyclic compounds with polar groups. Several studies have examined mild steel corrosion inhibitors in acidic environments(112)

1.13 Cancer and Anti-cancer:

According to a research from the World Health Organization (WHO), more than 13 million people would die from cancer worldwide in 2030. According to estimates, before the age of 75, one in five persons may develop cancer.The majority of malignancies are distinguished by an uncontrolled proliferation of cells that lack differentiation as a result of the regulation of crucial enzymes and other proteins regulating cell division and proliferation(113)**.** Scientists have been interested in nitrogen-containing heterocyclic for a long time because of its diverse structural makeup and biological significance. The most recent advancements in nitrogen-containing heterocyclic compounds as prospective cancer chemotherapeutics are covered in the current paper. A nitrogen heterocyclic is present in almost 60% of distinct small compounds(114). Madia et al. (2021) designed and synthesized pyrimidine-based analogues and evaluated their anticancer activity against some cancer cell lines. Each of synthesized compounds showed excellent anticancer activity. Among them, compound **3** showed excellent activity (115).



Figure(1-11) anticancer compounds containing pyrimidine ring

1.14.Aim of the Study:

1.Synthesis and characterization of new heterocyclic compounds derivatives and this include:

a-Synthesis of new azo compounds and chalcon
b-Synthesis of new five membern heterocyclic compounds (pyrazol,isoxazol)
c-Synthesis of new six membern heterocyclic compounds (oxazine,thiazin)
d-Synthesis of new seven member heterocyclic compounds (diazipen,oxazipen,thiazepin)
e- Synthesis of new schiff base from thiadiazole.

2. Testing biological activity for some of the synthesized compounds on different microorganisms.

3. Study the anti- corrosion for some of the prepared compounds.
4. Study the anti- cancer for some of the prepared compounds.

2.Experimental and Methods

2.1. Chemicals:

Table (2-1): Chemicals and their Suppliers:

Chemicals	Supplier	Purity%
p-amino benzoic acid	CDH	98
o-amino phenole	Merk	99
Benzaldehyde	CDH	98
Dichloromethane	CDH	99
Dimethyl sulfoxide	BDH	99
3,4-dichlorobenzaldehyde	CDH	98
2,4-dihydroxy acetophenone	Sigma-Aldrich	98
4-dimethylaminobenzaldehyde	CDH	99
Ethanol	Sigma-Aldrich	99.3
Glacial acetic acid	Sigma-Aldrich	98
Hydrazine hydrate	CDH	99
Hydrazin hydrochloride	Merk	99
Hydrochloric acid	CDH	37
4-hydroxy acetophenone	Sigma-Aldrich	98
p-methoxy benzaldehyde	CDH	98
3-methoxy-4-hydroxy benzaldehyde	CDH	99
4-Nitrobenzaldehyde	Merk	99
Pheniline diamine	CDH	99.8
Potassium hydroxide	BDH	99
Sodium hydroxide	CDH	80
Sodium nitrate	CDH	37
Thiocemicarbazide	BDH	98
Thio urea	Merk	99
o-thio aniline	Merk	98
Urea	Sigma-Aldrich	99

2.2 Instruments:

1-Melting points were verified by hot stage SMP30 melting point apparatus.

2-Infrared spectra were verified by Fourier Transform infrared SHIMADZU (8400) (FTIR) infrared spectrophotometer , KBr disc was performed by Babylon University .

3- ^{1}H-NMR Spectra and ^{13}C-NMR Spectra was verified on a Bruker (AC 400) NMR spectrometer, working at(400 MHz) for ^{1}H-NMR and (100MHz) for ^{13}C-NMR. All chemical shifts (δ) are reported in ppm relative to tetramethylsilane (TMS) as references (δ=0.0 ppm)

4- CHNS was used for characterization of the prepared compounds .

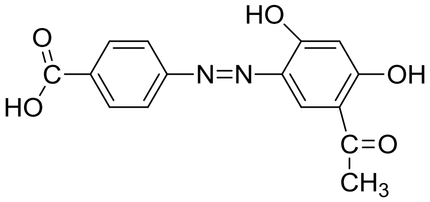
5-The biological activity was performed by Microbiology Unite , Babylon Hospital

6- The corrosion was verified by potentiometer and was perfomed by Baghdad University .

7-The anti-cancer was measured in an National Cell Bank of Iran (Pasteur Institute, Iran).

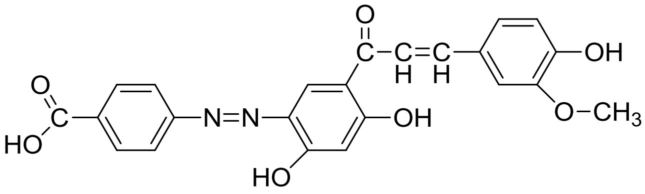
2.3. Methods :

2.3.1. Synthesis of Compound [S1](116):



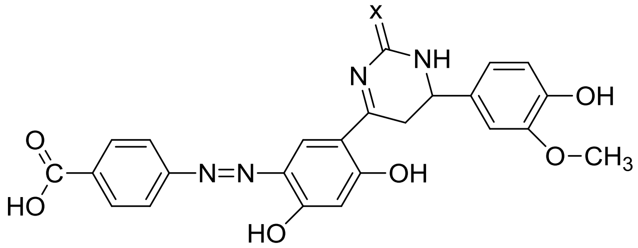
P-Aminobenzoicacid (0.01mole, 1.37g) was dissolved in 17ml distill water and 3ml HCl at a temperature of (0-5)^{0}C. The solution was then dropwise added (0.01 mole, 0.69g) of $NaNO_2$ dissolved in (10 ml) distilled water for (15) minutes. The diazonium salt were add dropwise to the coupling component solution, which is maded using (0.01mole,1.520g) 2,4-dihydroxy acetophenone in ethanol with (1 g) sodium hydroxide in (10ml) distill water. after that, the precipitate was filtered and washed with water ,the precipitate was recrystallized by absolute ethanol .

2.3.2. Synthesis of Compound[S2](117):



(0.01 mole ,3 gm) of compound [S1] is dissolve in absolute ethanol and 30% NaOH in mixture of ethanol and distilled water, was added as dropwise to [S1] compound with stirrer after that (0.01 mole ,1.5 gm)of aldehyde (4-hydroxy-3-methoxy benzaldehyde) was added ,the mixtur stirror for 6 hours and the the sediment was left in the refrigerator over night.

2.3.3. Synthesis of Compound [S3,S4](19,20):



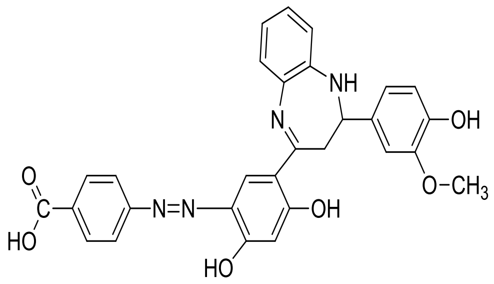
x:o,s

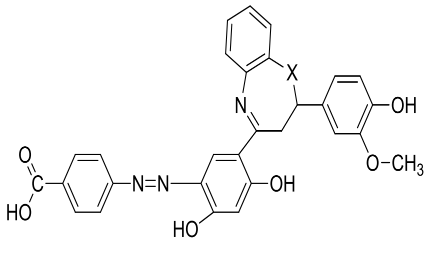
A mixture of chalcone [S2] (0.01 mol ,3 gm),urea/ thiourea (0.01 mol,0.6gm /0.76 gm) were dissolved in (30 mL) of absolute ethanol and sodium hydroxide solution 10% (5 mL) was stirred for 4hrs, after that it was transferred into 20 mL of ice water with continuous stirring for 1 hrs, then left overnight.

The precipitate made was filtered, washed and recrystallized by ethanol.

2.3.4. Synthesis of Compound [S5-S7](118)

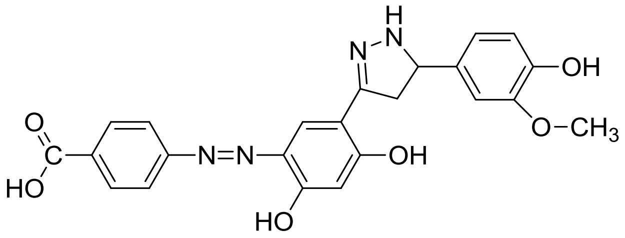
A mixture of chalcone [S2] (0.01 mole,3 gm),was mixed with (0.01 mole,1.08 gm, 1.09 gm, 1.25 gm) of compounds(phenylenediamine,orthoaminophenol,orthothioaminophenol) and dissolved in(30 ml) absolute ethanol and few drops of sodium hydroxide (10 %) was refluxed 3-4 hrs, with continuous stirring ,after that addition of (1 ml) of acetic acid and again reflexed for 3-4 hrs then kept in refrigerator for 24 hrs. The product was filtered ,washed and recrystallized by absolute ethanol·





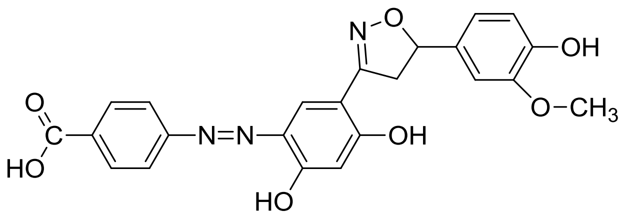
x:o,s

2.3.5 Synthesis of Compounds [S8]$^{(119)}$



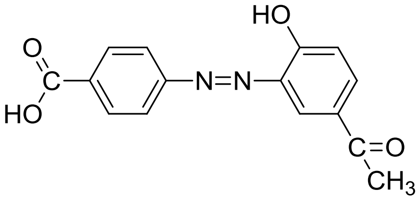
Chalcone [S2] (0.01 mole,3 gm) were dissolved in ethanol 99% (10ml) and (1.0gm) NaOH and refluxed with excess of ($NH_2NH_2.H_2O$) (5-10ml) for 12hrs.at 75°C.$^{(124)}$

2.3.6 Synthesis of Compound [S9]$^{(119)}$



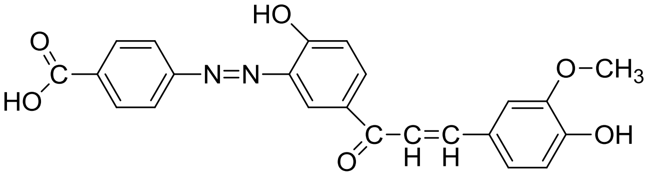
A mixture of chalcone (0.01 mole,3 gm) hydroxylamine hydrochloride (0.025g in 0.25 mL of water) in 5 mL ethanol 99% was added to sodium hydroxide solution (0.05g NaOH in 0.5mL of water). The mixture was refluxed in water bath for 8hr, cooling to room tempreture then freezing overnight, the mixture was concentrated by destilating out the solvent under reduced pressure and then poured into crushed ice.

2.3.7 Synthesis of Compounds [S10](116)



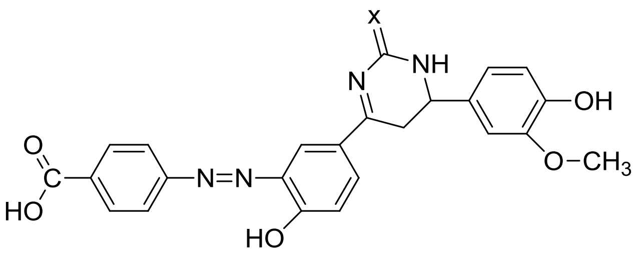
At a temperature of 0 to 5 ^{0}C, (0.01mole, 1.37gm) of *p*-aminobenzoicacid was dissolved in 17ml of distiller's water and 3ml of HCl. The solution was then added dropwise for 15 minutes using (0.01mole ,0.69g) of $NaNO_2$ dissolved in 10 ml of distilled water. The coupling component solution was created by mixing (0.01mole, 1.36gm of 4-hydroxyacetophenone in ethanol with 1 g of sodium hydroxide in 10ml of distill water. The diazonium salt was added dropwise to this mixture. The precipitate was then filtered, washed with water, and recrystallized using 99% ethanol.

2.3.8 Synthesis of Compound [S11](117)



(0.01 mole ,3 gm) of compound [S10] is dissolve in absolute ethanol and 30% NaOH in mixture of ethanol and distilled water, was added as dropwise to chalcon with stirrer after that (0.01 mole ,1.5 gm)of aldehyde (4-hydroxy-3-methoxybenzaldehyde) was added ,the mixtur stirror for 6 hours and the the sediment was left in the refrigerator over night

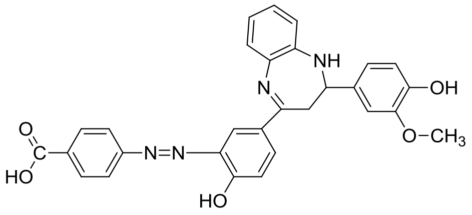
2.3.9 Synthesis of Compound [S12,S13](19-20):

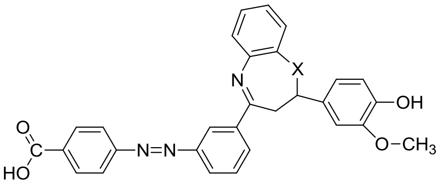


x:o,s

A mixture of chalcone [S11] (0.01 mol ,3 gm),urea/ thiourea (0.01 mol,0.6gm /0.76 gm) were dissolved in (30 mL) of absolute ethanol and sodium hydroxide solution 10% (5 mL) was stirred for 4hrs, after that it was transferred into 20 mL of ice water with continuous stirring for 1 hrs, then left overnight. The precipitate made was filtered, washed and recrystallized by ethanol.

2.3.10 Synthesis of Compound [S14-S16]⁽¹¹⁸⁾

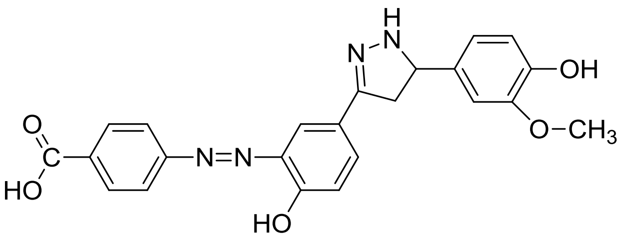




x=o,s

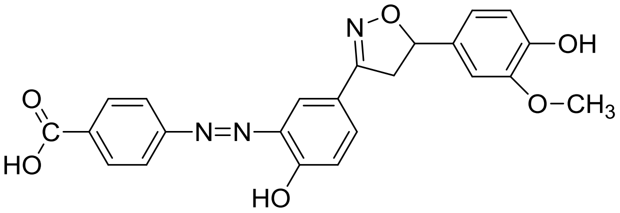
A mixture of chalcone [S11] (0.01 mole,3 gm),was mixed with (0.01 mole,1.08 gm, 1.09 gm, 1.25 gm) of compounds(phenylenediamine,orthoaminophenol,orthothio aminophenol) respectively and dissolved in(30 ml) absolute ethanol and few drops of sodium hydroxide (10 %) was refluxed 3-4 hrs, with continuous stirring ,after that addition of (1 ml) of acetic acid and again reflexed for 3-4 hrs then kept in refrigerator for 24 hrs. The product was filtered ,washed and recrystallized by absolute ethanol(123).

2.3.11 Synthesis of Compounds [S17]⁽¹¹⁹⁾



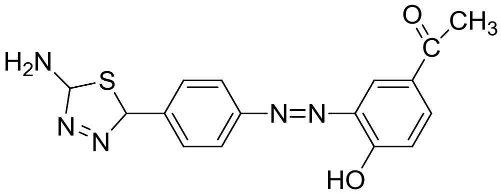
Chalcone [S11] (0.01 mole,3 gm) were dissolved in ethanol 99% (10ml) and (1.0gm) NaOH and refluxed with excess of ($NH_2NH_2.H_2O$) (5-10ml) for 12hrs.at 75°C.(124)

2.3.12 Synthesis of Compound [S18](119)



A mixture of chalcone [S11] (0.01 mole,3gm), hydroxylamine hydrochloride (0.025g in 0.25 mL of water) in 5 mL ethanol 99% was added to sodium hydroxide solution (0.05g NaOH in 0.5mL of water). The mixture was refluxed in water bath for 8hr, cooling to room temperature then freezing overnight, the mixture was concentrated by destilating out the solvent under reduced pressure and then poured into crushed ice

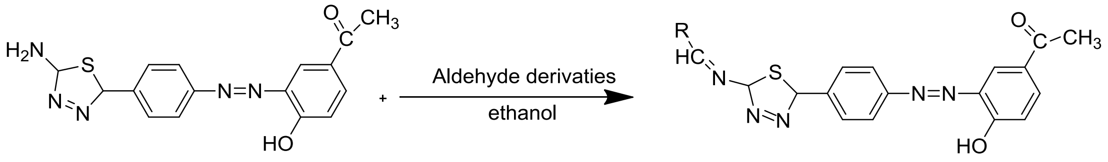
2.3.13 Synthesis of Compound [S19](120)



(0.01mole, 2.58g) of compound [S10] and (0.01 mole, 0.91g) thiosemicarbazide were dissolved in (8 ml) $POCl_3$, then was refluxed for (4) hours, after that (40 ml) distilled water was added and refluxed for (4)hours ,then added (4 g) potassium hydroxide in (40ml) distilled water. Then filtered and recrystallized by absolute ethanol

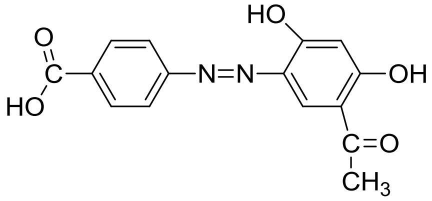
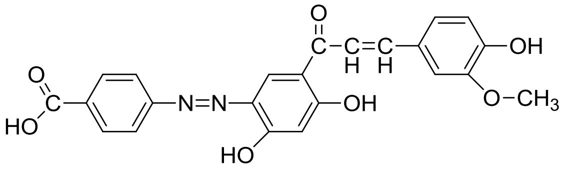
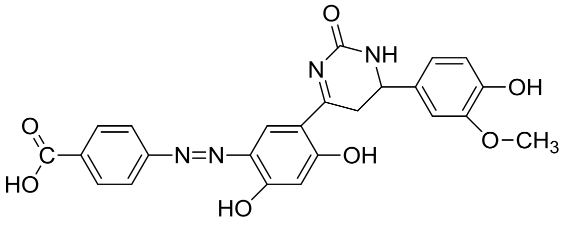
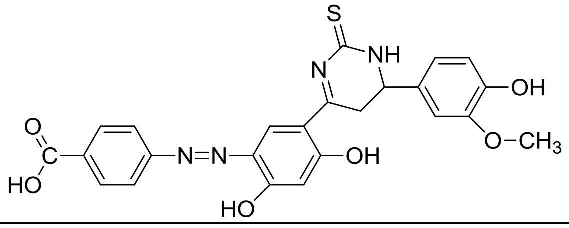
2.3.14 Synthesis of Compounds [S20-S25](121)

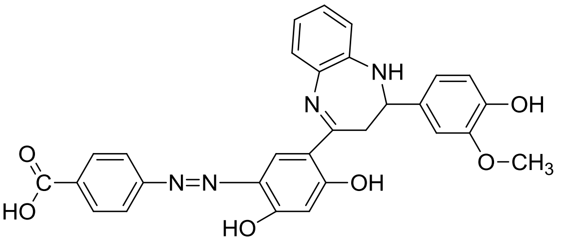
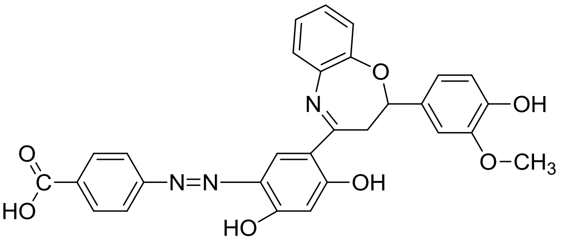
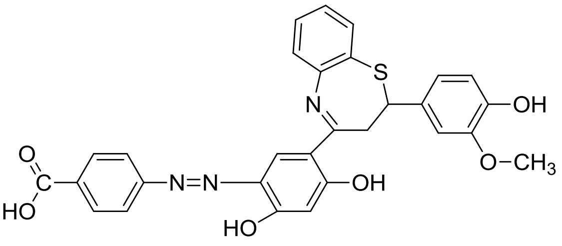
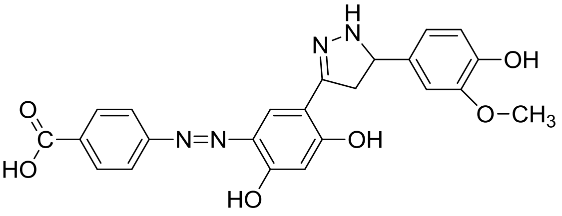
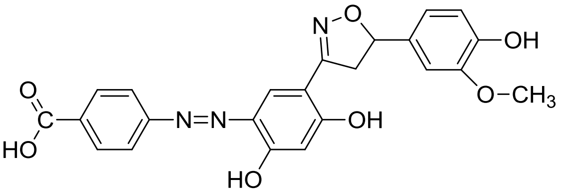
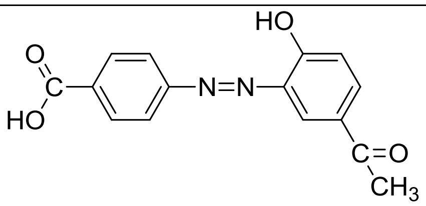
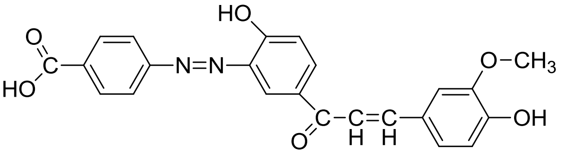
0.01 mol of thiadiazoleamine reacts with (vanillin, benzaldehyde, *p*-methoxybenzaldehyde,dimethylaminobenzaldehyde,*p*-nitrobenzaldehyde, dichlorobenzaldehyde) to produce basic compounds (Schiff Base) after dissolving it in absolute ethanol with the presence of a few drops of glacial acetic acid.

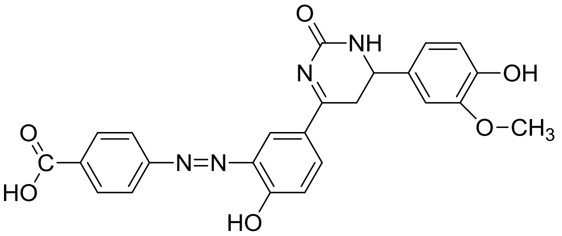
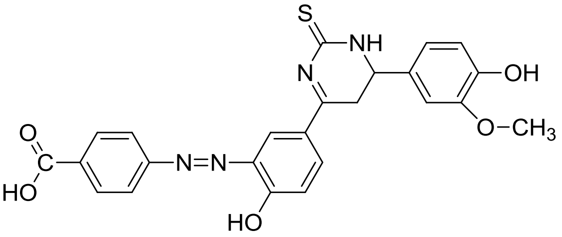
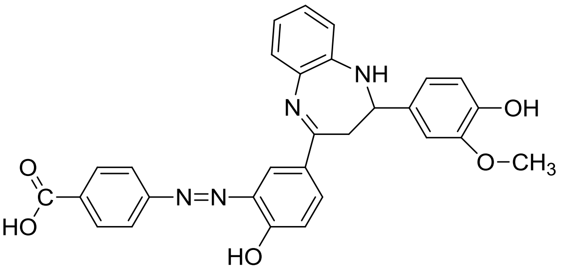
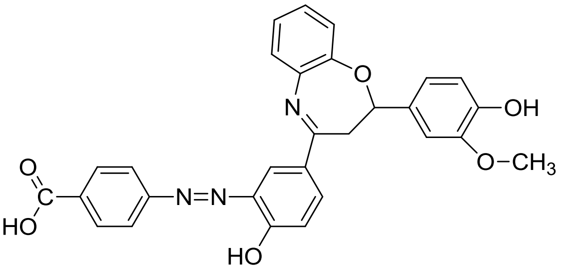
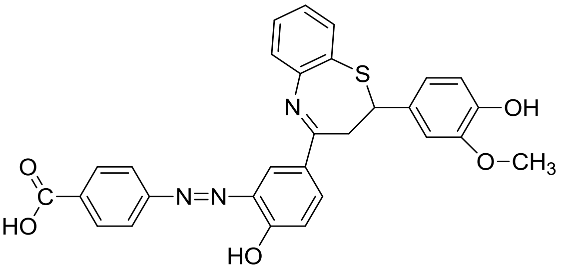
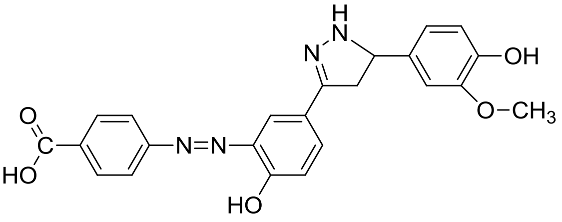


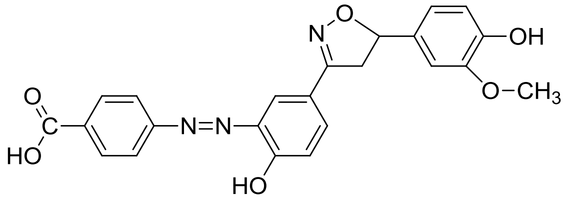
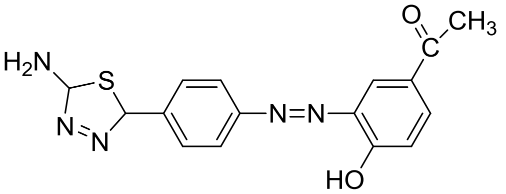
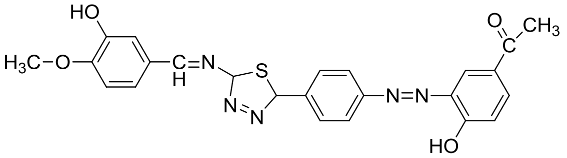
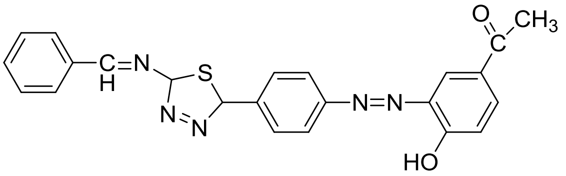
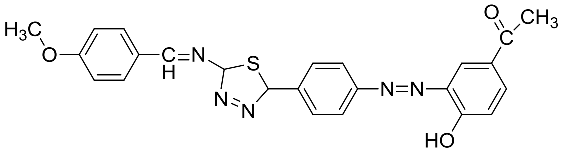
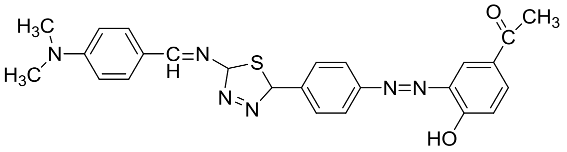
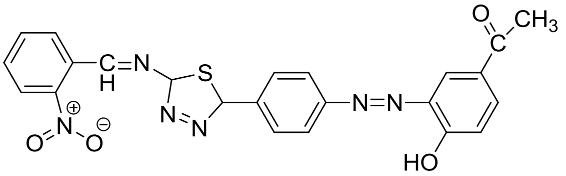
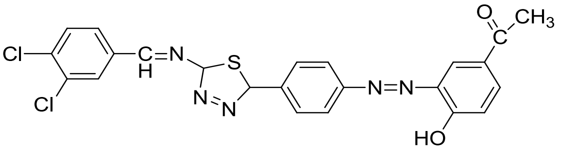
R= Vanillin,benzaldehyde,*p*-methoxy benzaldehyde,dimethylaminobenzaldehyde,*p*-nitrobenzaldehyde,dichlorobenzaldehyde

Table (2-2) : Chemical Structure and Name of Preparation Compounds [S1-S25]:

comp. no.	compound structure	compound name
[S1]		4-((5-acetyl-2,4-dihydroxyphenyl)diazenyl) benzoic acid
[S2]		E)-(2,4-dihydroxy-5-((Z)-3-(4-hydroxy-3-))-4 methoxyphenyl)acryloyl)phenyl)diazenyl)benzoic acid
[S3]		dihydroxy-5-(6-(4-hydroxy-3--2,4))-4 methoxyphenyl)-2-oxo-1,2,5,6-tetrahydropyrimidin-4-yl)phenyl)diazenyl)benzoic acid
[S4]		dihydroxy-5-(6-(4-hydroxy--2,4))-4 3-methoxyphenyl)-2-thioxo-1,2,5,6-tetrahydropyrimidin-4-yl)phenyl)diazenyl)benzoic acid

[S5]		dihydroxy-5-(2-(4-hydroxy--2,4))-4 3-methoxyphenyl)-1H-benzo[b][1,4]diazepin-4-yl)phenyl)diazenyl)benzoic acid
[S6]		dihydroxy-5-(4-(4-hydroxy--2,4))-4 3-methoxyphenyl)-4,5-dihydrobenzo[b][1,4]oxazepin-2-yl)phenyl)diazenyl)benzoic acid
[S7]		dihydroxy-5-(4-(4-hydroxy--2,4))-4 3-methoxyphenyl)-4,5-dihydrobenzo[b][1,4]oxazepin-2-yl)phenyl)diazenyl)benzoic acid
[S8]		dihydroxy-5-(5-(4-hydroxy--2,4))-4 3-methoxyphenyl)-1H-pyrazol-3-yl)phenyl)diazenyl)benzoic acid
[S9]		dihydroxy-5-(5-(4--2,4))-4 hydroxy-3-methoxyphenyl)-4,5-dihydroisoxazol-3-yl)phenyl)diazenyl)benzoic acid
[S10]		acetyl-2--5))-4 hydroxyphenyl)diazenyl)benzoic acid
		hydroxy-5-((E)-3-(4-hydroxy--2))-4 3-methoxyphenyl)acryloyl)phenyl)diazenyl)benzoic acid

[S11]		
[S12]		hydroxy-5-(6-(4-hydroxy-3--2))-4 methoxyphenyl)-2-oxo-1,2,5,6-tetrahydropyrimidin-4-yl)phenyl)diazenyl)benzoic acid
[S13]		hydroxy-5-(6-(4-hydroxy-3--2))-4 methoxyphenyl)-2-thioxo-1,2,5,6-tetrahydropyrimidin-4-yl)phenyl)diazenyl)benzoic acid
[S14]		hydroxy-5-(2-(4-hydroxy-3--2))-4 methoxyphenyl)-2,3-dihydro-1H-benzo[b][1,4]diazepin-4-yl)phenyl)diazenyl)benzoic acid
[S15]		hydroxy-5-(2-(4-hydroxy-3--2))-4 methoxyphenyl)-2,3-dihydrobenzo[b][1,4]oxazepin-4-yl)phenyl)diazenyl)benzoic acid
[S16]		hydroxy-5-(2-(4-hydroxy-3--2))-4 methoxyphenyl)-2,3-dihydrobenzo[b][1,4]thiazepin-4-yl)phenyl)diazenyl)benzoic acid
[S17]		hydroxy-5-(5-(4-hydroxy-3--2))-4 methoxyphenyl)-4,5-dihydro-1H-pyrazol-3-yl)phenyl)diazenyl)benzoic acid

[S18]		hydroxy-5-(5-(4-hydroxy-3--2))-4 methoxyphenyl)-4,5-dihydroisoxazol-3-yl)phenyl)diazenyl)benzoic acid
[S19]		amino-2,5-dihydro-1,3,4--5)-4))-3)-1 thiadiazol-2-yl)phenyl)diazenyl)-4-hydroxyphenyl)ethanone
[S20]		hydroxy-3-((4-(5-((3-hydroxy-4--4)-1 methoxybenzylidene)amino)-2,5-dihydro-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)phenyl)ethanone
[S21]		benzylideneamino)-2,5-)-5)-4))-3)-1 dihydro-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)-4-hydroxyphenyl)ethanone
[S22]		hydroxy-3-((4-(5-((4--4)-1 methoxybenzylidene)amino)-2,5-dihydro-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)phenyl)ethanone
[S23]		-4))-5)-4))-3)-1 dimethylamino)benzylidene)amino)-) 2,5-dihydro-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)-4-hydroxyphenyl)ethanone
[S24]		hydroxy-3-((4-(5-((2--4)-1 nitrobenzylidene)amino)-2,5-dihydro-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)phenyl)ethanone
[S25]		-3,4))-5)-4))-3)-1 dichlorobenzylidene)amino)-2,5-dihydro-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)-4-hydroxyphenyl)ethanone

Table(2-3):Physical Properties to the Synthesized Compounds :

Compound Number	M.P (^{0}C)	Yield (%)	Color	Molecular Weight (g\mole)	Molecular formula
[S1]	168-170	93	orange-red	300	$C_{15}H_{12}O_5N_2$
[S2]	149-150	91	Dark red	418	$C_{23}H_{18}O_7N_2$
[S3]	Oily	92	black	475	$C_{24}H_{19}O_7N_4$
[S4]	60-63	89	light red	492.5	$C_{24}H_{19}O_6N_4S$
[S5]	Oily	90	black	520	$C_{29}H_{23}O_6N_4$
[S6]	Oily	74	dark red	524	$C_{29}H_{23}O_7N_3$
[S7]	60-62	80	dark orange	541	$C_{29}H_{23}O_6N_3S$
[S8]	69-72	85	orange	445	$C_{23}H_{17}O_6N_4$
[S9]	67-69	88	black	464	$C_{23}H_{16}O_7N_3$
[S10]	165-167	90	orange	283	$C_{15}H_{11}O_4N_2$
[S11]	245-247	92	darck-red	401	$C_{23}H_{18}O_6N_2$
[S12]	195-197	84	darck	458	$C_{24}H_{20}O_6N_4$
[S13]	80-83	80	light red	475	$C_{24}H_{20}O_5N_4S$
[S14]	200-202	85	black	503	$C_{29}H_{25}O_5N_4$
[S15]	Oily	74	black	507	$C_{29}H_{23}O_6N_3$
[S16]	65-67	76	Pale orange	524	$C_{29}H_{23}O_5N_3S$
[S17]	62-64	75	orange	432	$C_{21}H_{20}O_5N_4$
[S18]	Oily	60	black	434	$C_{23}H_{19}O_6N_3$
[S19]	133-135	93	brown	341	$C_{16}H_{15}O_2N_5S$
[S20]	265-267	90	black	475	$C_{24}H_{21}O_4N_5S$
[S21]	267-269	94	Pale red	429	$C_{23}H_{19}O_2N_5S$
[S22]	222-224	89	red	459	$C_{24}H_{21}O_3N_5S$
[S23]	226-228	87	black	472	$C_{25}H_{24}O_2N_6S$
[S24]	109-111	88	black	474	$C_{23}H_{18}O_4N_6S$
[S25]	277-279	90	red	497	$C_{23}H_{17}O_2N_5SCl_2$

3.Results and Discussion

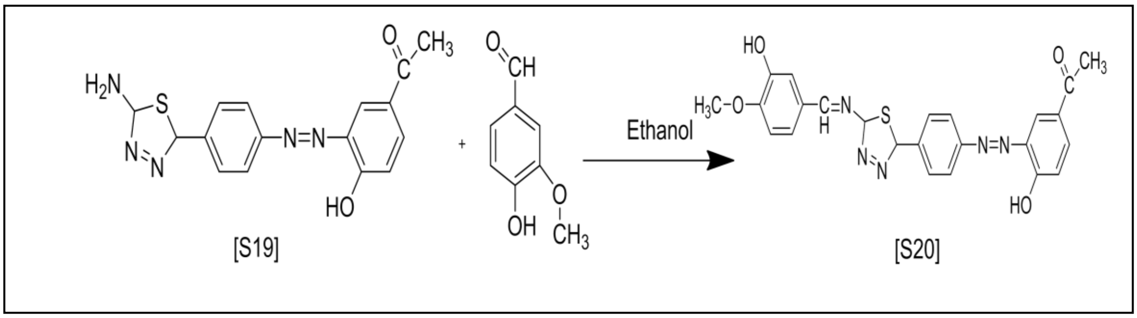
3.1 Synthesis of Azo Compound [S1] :

The first reaction involve reaction of *p*-aminobenzoic acid as nucleophile (rich in electron) with nitrous ion to form diazonium salt, after (15 min) this diazonium reaction with coupling compound, The reaction should be carried out at (0-5°C) with continuous stirring to form azo dye.

The FTIR (ν, cm^{-1}) spectrum exhibited absorption band at (1604) cm^{-1} for N=N and disappearance absorption band at (3363,3473) cm^{-1} for NH_2, O-H (3267),C-H Ar (3101), C=O carboxylic acid (1691), $C{=}O_{keton}$ (1650), N=N (1604) .

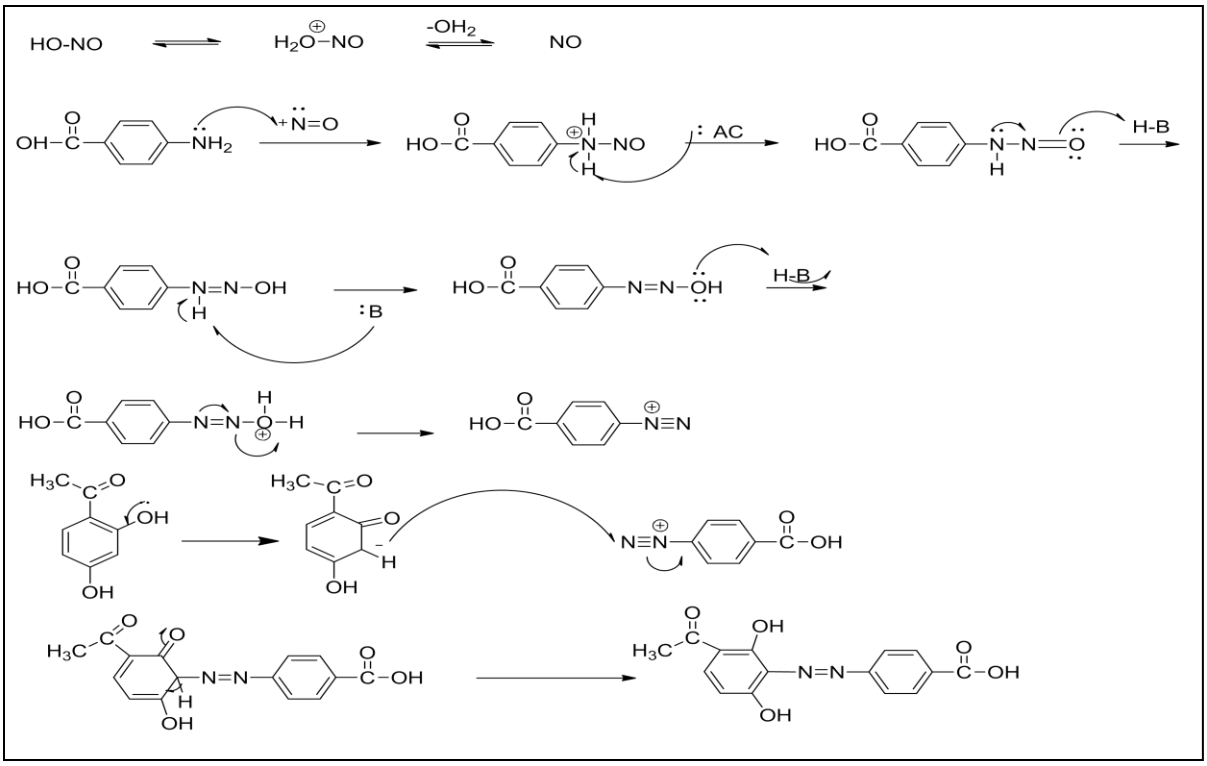
^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 5.6 for (H,OH) and signal at 2.6 for (H,CH_{alpha}), (6H,Ar-H) : 6.85-8.08.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at 26.72 for,$C\text{-}H_{alpha}$, (118-132) for (12C,Ar-C),(167.26) for $C{=}O_{\text{carboxylic acid}}$,(196.57) for $C{=}O_{\text{ketone}}$.



Equation 3-1: Synthesis of Compound [S1]

The mechanism of azo synthesis is shown below(122)



Scheme(1) The mechanism of azo synthesis

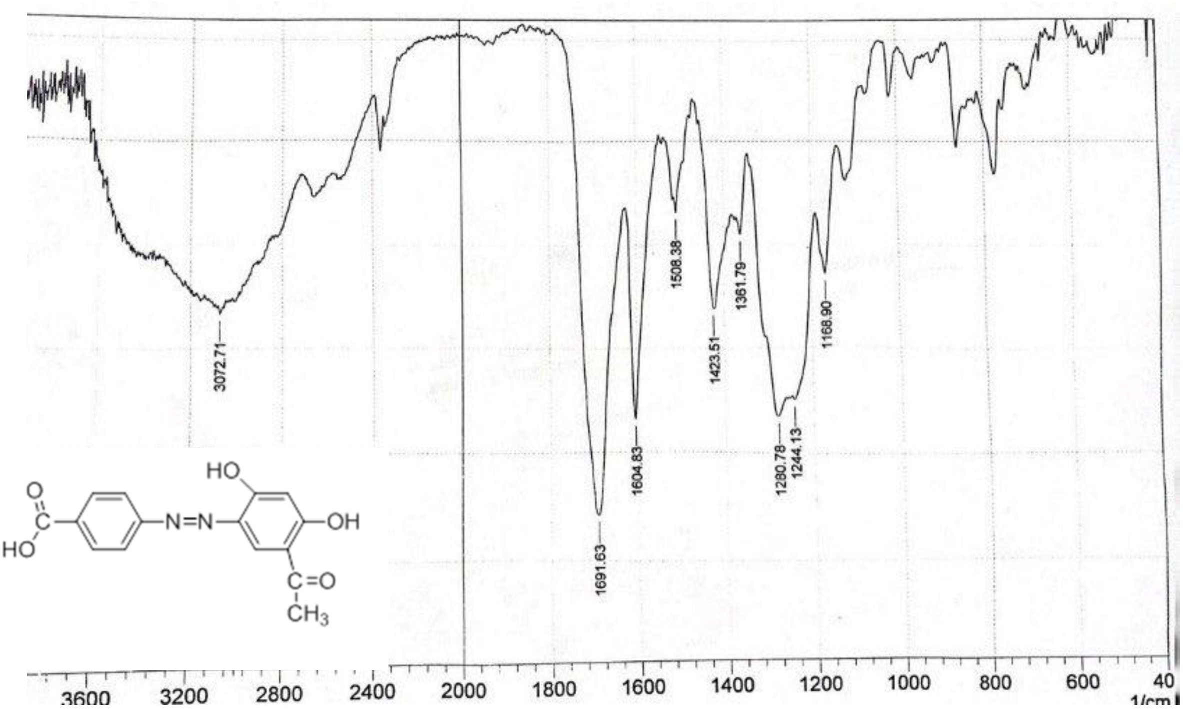
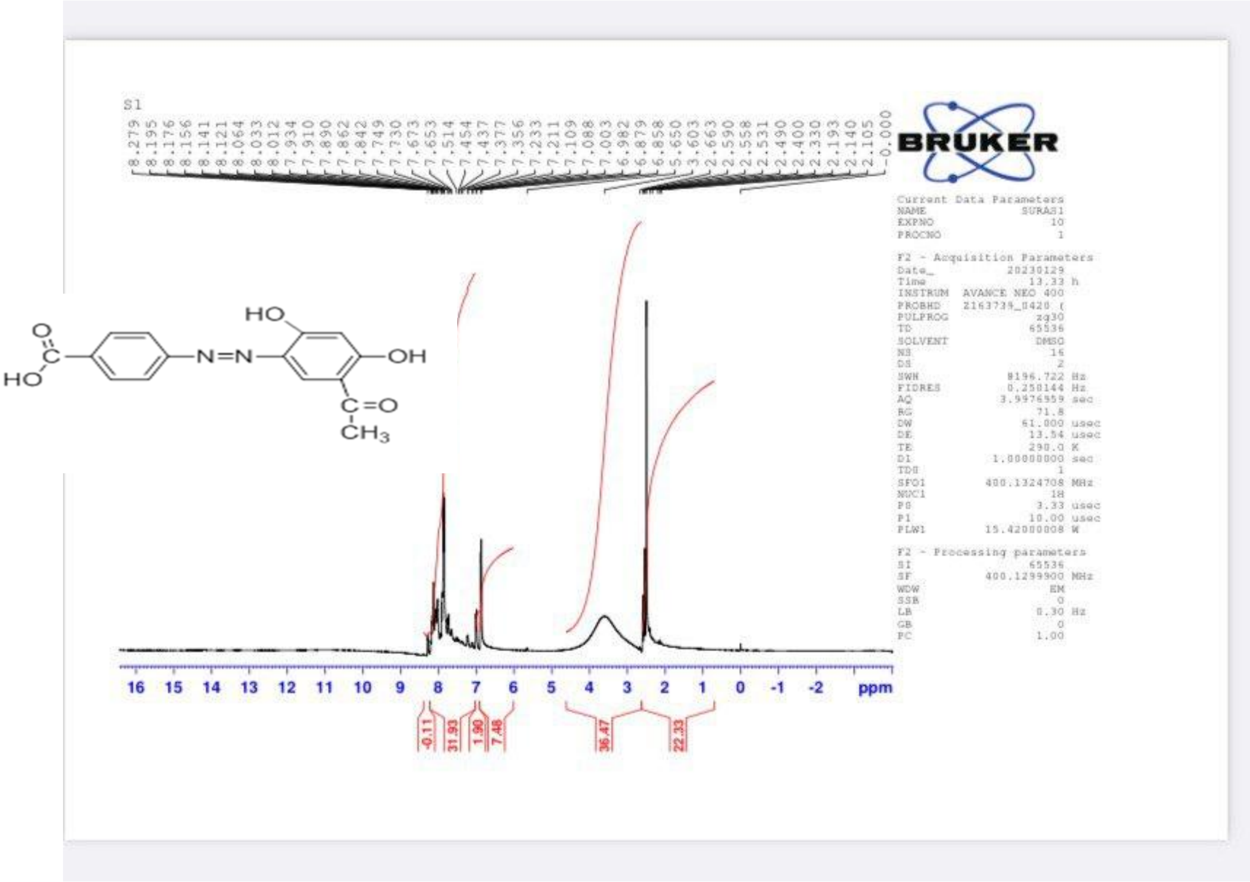


Figure (3-2)FT-IR Spectrum of compound[S1]



Figure(3.3) ^{1}H-NMR of compound [S1]

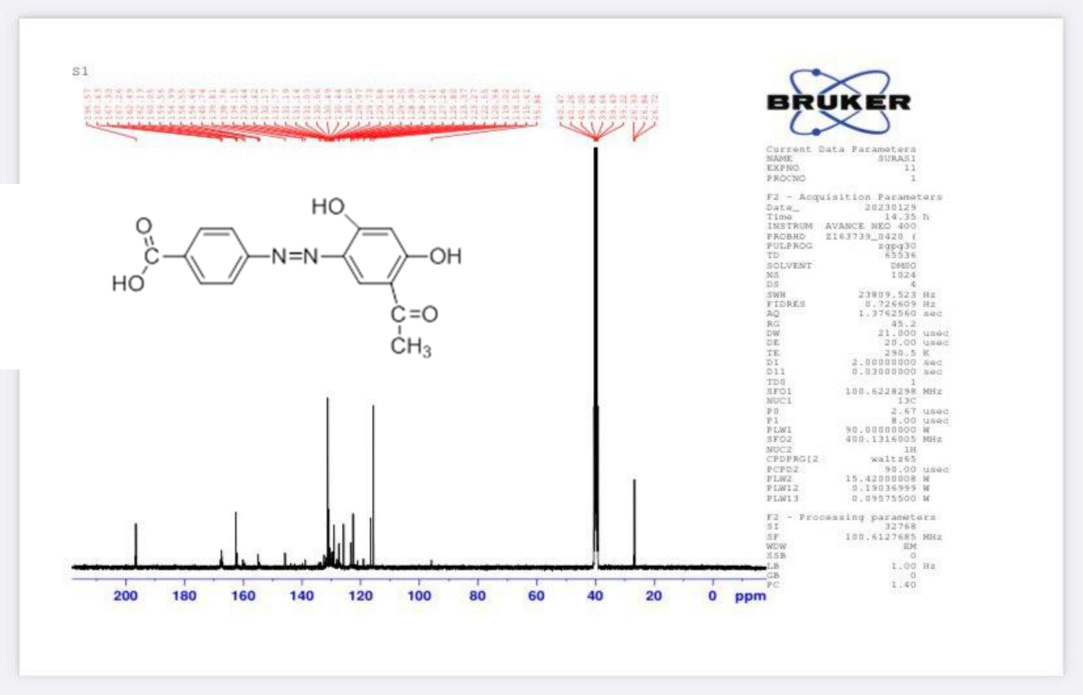


Figure (3-4)^{13}C-NMR of compound [S1]

3.2 Synthesis of compound[S2]:

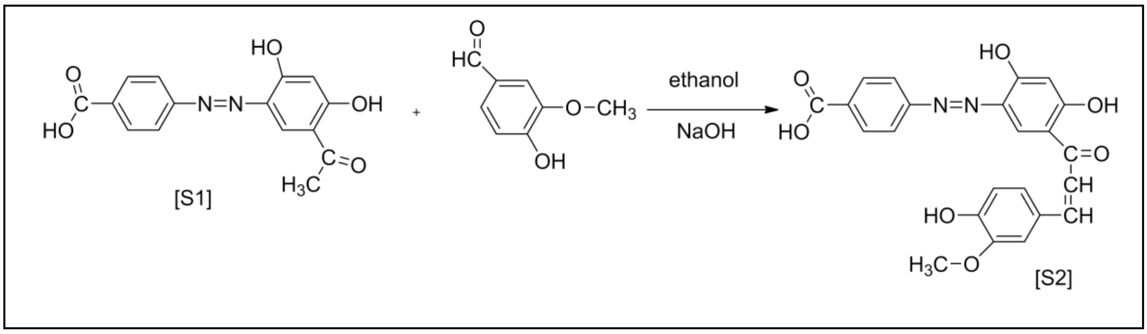
Chalcone [S2] synthesized from azo dye(4-((5-acetyl-2,4-dihydroxyphenyl)diazenyl) benzoic acid [S1] with aldehyde such as vanillin(4-hydroxy-3-methoxy benzaldehyd) in presence of ethanol as solvent and (10% NaOH ,dissolved in mix of ethanol and distill water as catalyst, When the hydroxyl group in the base accepts the hydrogen atom in the carbonyl group's alpha position, an aldol condensation process takes place, resulting in the release of a water molecule

The FTIR(v, cm^{-1}) spectrum of compound [S2] exhibited absorption band at (1280) cm^{-1} for O-CH_{ether} , (1620) cm^{-1} for C=C $_{alpha}$, C-H $_{Ar}$ (3173) , C=O $_{carboxylic\ acid}$ (1699), C=O_{ketone} (1683), N=N (1604).

^{1}H-NMR(100MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 5.1 for (H,OH) and signal at 6.8-8.2 for (10H, CH $_{Arom}$) ,

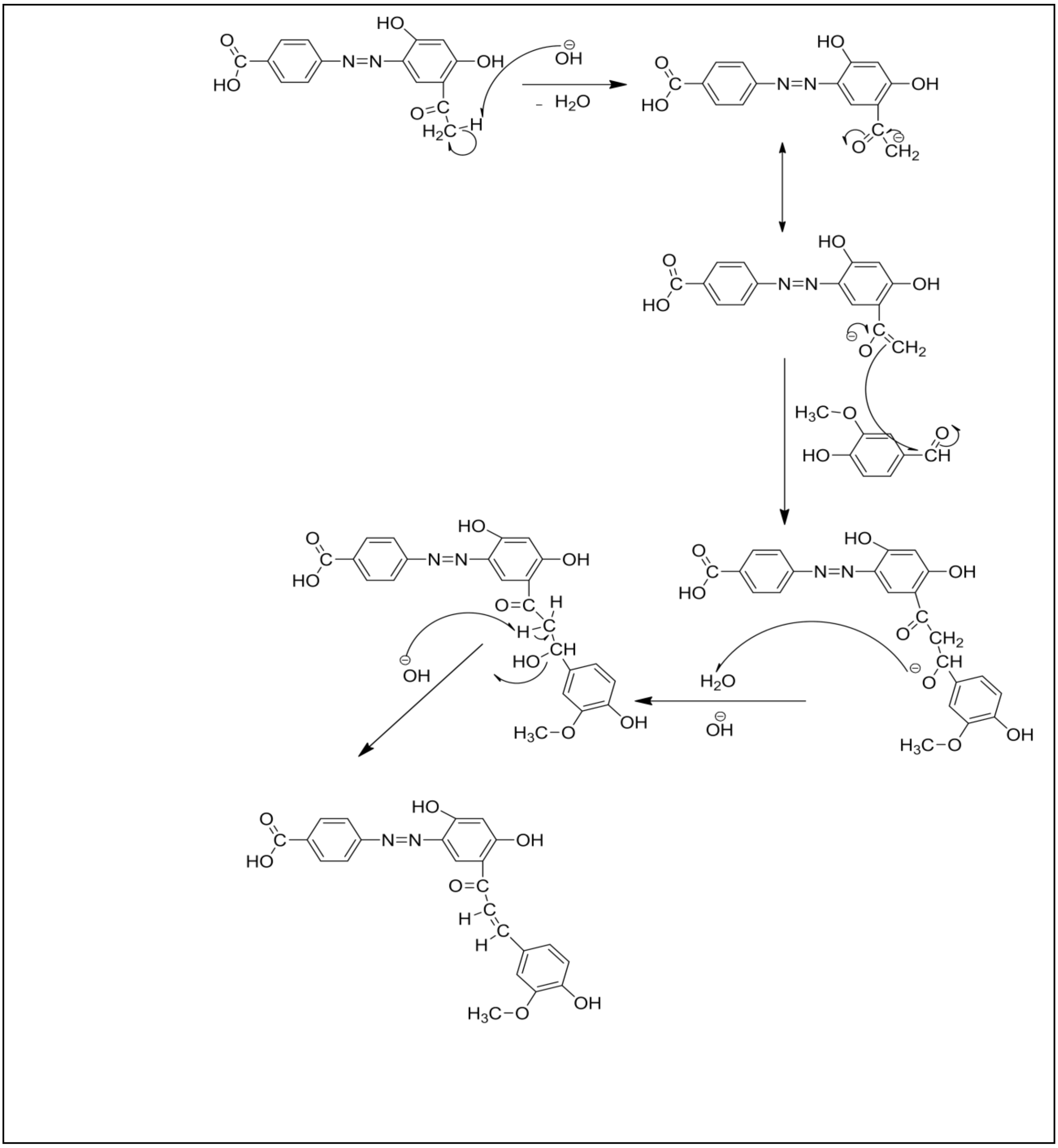
.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at (110-131) for 18C- Ar-C and signal at 55.97 for, C-H $_{alpha}$, (196) for C=O $_{Ketone}$,(168) for C=O $_{Carboxylic}$



Equation 3-2: Synthesis of Compound [S2]

The mechanism of chalcone synthesis is shown below(123)



Scheme (2)The mechanism of chalcone synthesis:

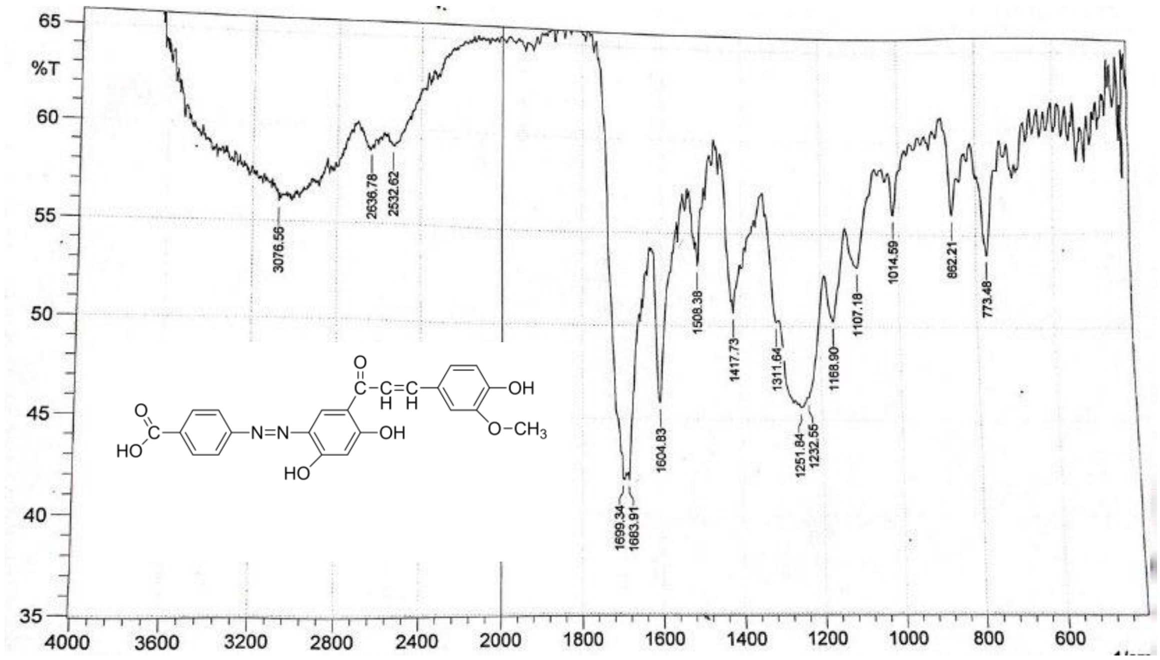
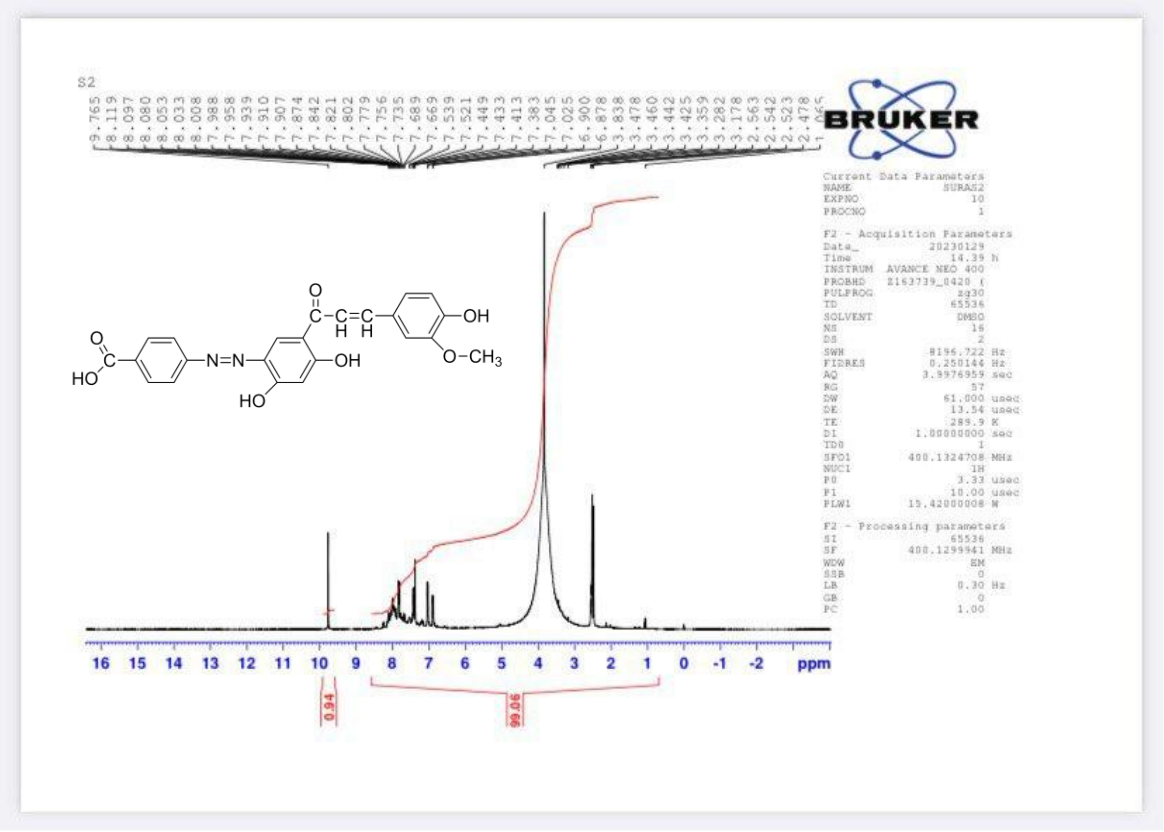


Figure (3-5) FT-IR Spectrum of compound[S2]



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PROCNO                 1

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NS                    16
DS                     2
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FIDRES          0.250144 Hz
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RG                    57
DW                61.000 usec
DE                 13.54 usec
TE                 289.9 K
D1            1.00000000 sec
TD0                    1
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NUC1                  1H
P0                  3.33 usec
P1                 10.00 usec
PLW1         15.42000008 W

F2 - Processing parameters
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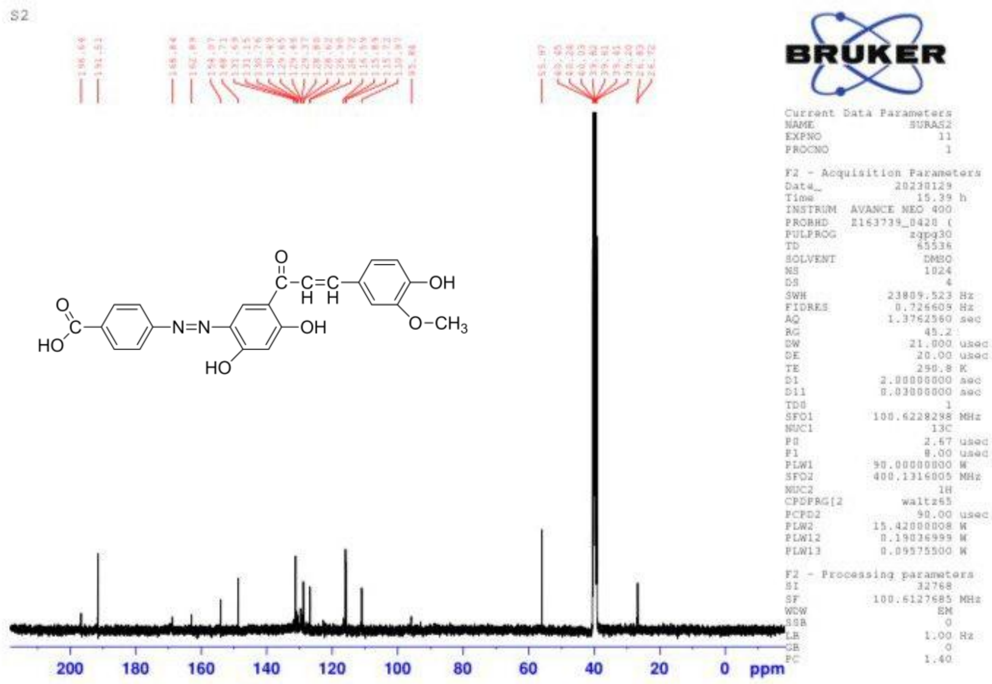
Figure (3-6) ^{1}H-NMR of compound [S2]



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DE                  20.00 usec
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TD0                     1
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NUC1                  13C
P0                   2.67 usec
P1                   8.00 usec
PLW1          90.00000000 W
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz65
PCPD2               90.00 usec
PLW2          15.42000008 W
PLW12          0.19036999 W
PLW13          0.09575500 W

F2 - Processing parameters
SI                  32768
SF            100.6127685 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

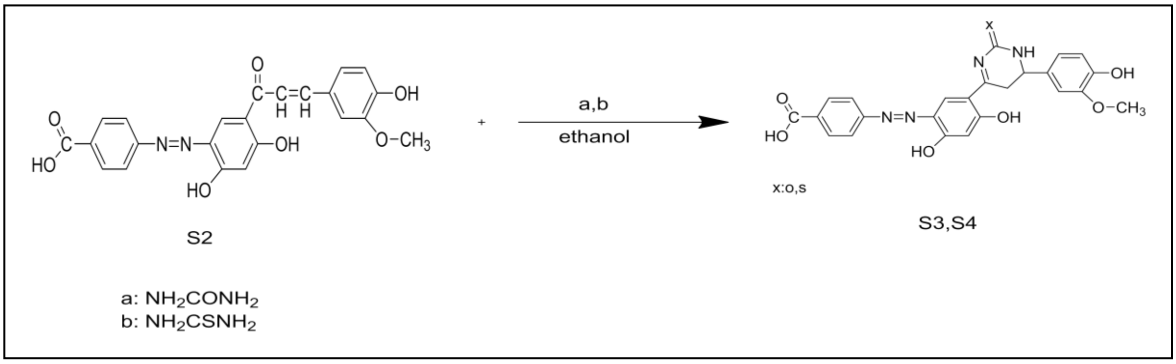
Figure (3-7) ^{13}C-NMR of compound [S2]

3.3 Synthesis of compound[S3,S4]:

(2-Oxo-pyrimidine, 2-thio-pyrimidine) These compounds were prepared from the reaction of (urea and thiourea) with ethanol in the presence of a few drops of sodium hydroxide with continuous stirring in an ice bath, and heterocyclic compounds with a hexagonal ring were produced when the chalcone interacted with urea and thiourea as a result of that a viscous compound, while a powdery compound was produced when it interacted with thiourea. After the amine group disappeared in the first reaction, it reappeared in compounds [S3,S4] (3331,3423) as a result of the interaction of the chalcone with urea and thiourea.

The FTIR(v, cm^{-1}) spectrum of compound [S3] exhibited absorption band at (3331) cm^{-1} for N-H , and disappearance absorption band at (1683) cm^{-1} for C=O $_{\text{ketone}}$, C-H $_{\text{Ar}}$ (3173), C=O $_{\text{carboxylic acid}}$ (1695),N=N (1595), C-OR (1282), C-H Ar (3070)

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 5.6 for (H,OH) and signal at 3.4 for (H, CH $_{\text{Arom}}$) ,8.4 for(H-NH$_{\text{endocyclic}}$) ,10.8 for (HCOOH),1.5 for (H-CH$_{\text{endocyclic}}$)



Equation 3-3: Synthesis of Compound [S3,S4]

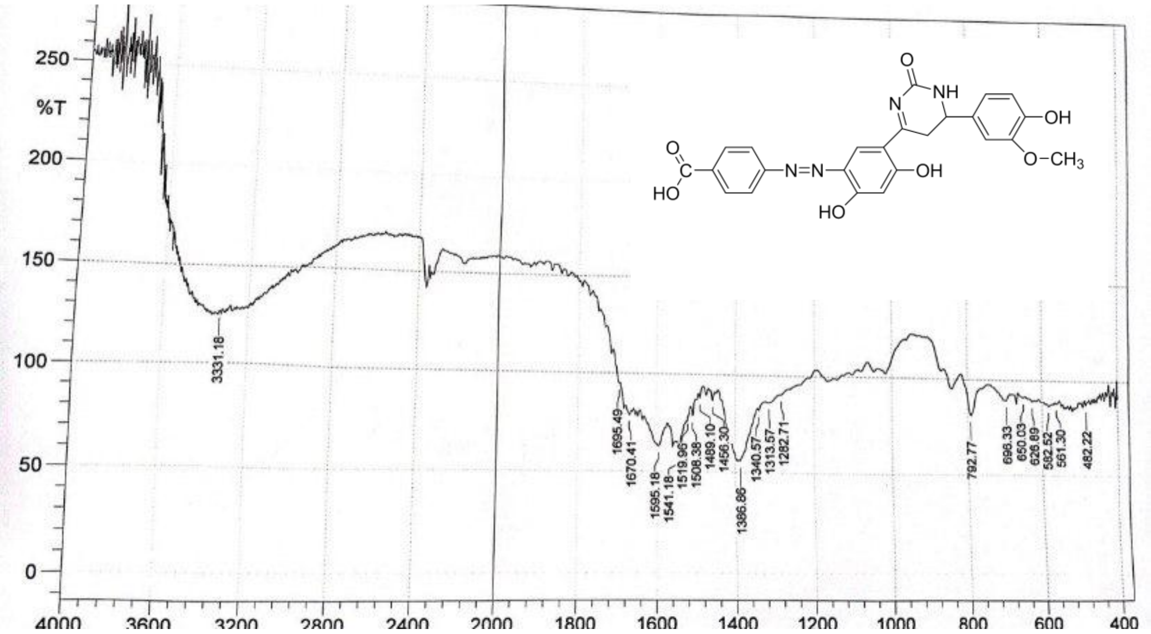
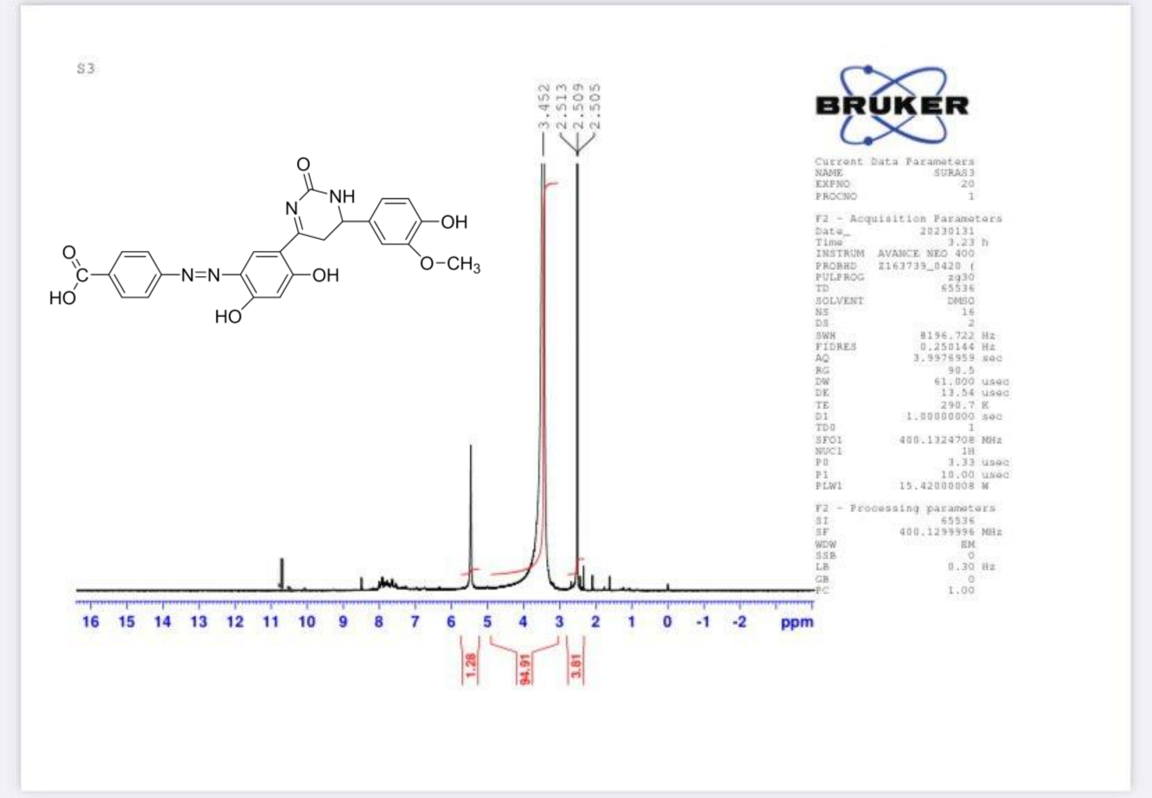


Figure (3-8) FT-IR Spectrum of compound[S3]



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PROCNO                 1

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PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ             3.9976959 sec
RG                  90.5
DW                61.000 usec
DE                 13.54 usec
TE                 290.7 K
D1            1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P0                  3.33 usec
P1                 10.00 usec
PLW1         15.42000008 W

F2 - Processing parameters
SI                 65536
SF           400.1299996 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

Figure (3-9) ^{1}H-NMR of compound [S3]

The FTIR (*v*, cm^{-1}): spectrum of compound [S4] exhibited absorption band at (3435) cm^{-1} for N-H , and disappearance absorption band at (1683) cm^{-1} for C=O $_{\text{keton}}$, C-H $_{\text{Ar}}$ (3045), C=O $_{\text{carboxylic acid}}$ (1683),N=N (1606), C=S(1178), C-OR (1284),C-H $_{\text{Ar}}$ (3045).

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 5.6 for (H,OH) and signal at (6.8-8.01) for (H, CH $_{\text{Arom}}$) ,8.12 for (H-NH$_{\text{endocyclic}}$) ,9.7 for (HCOOH),1.3 for(H-CH$_{\text{endocyclic}}$)

13C-NMR(400MHz,DMSO-d6) : (δ, ppm) : 18C,Ar-C (111-131), C=O $_{\text{carboxylic acid}}$ (167.8), C,OR :(148)$_{,}$ C,C-H$_{\text{alpha}}$ (56) ,C,C-H$_{\text{endocyclic}}$:(40.4) .

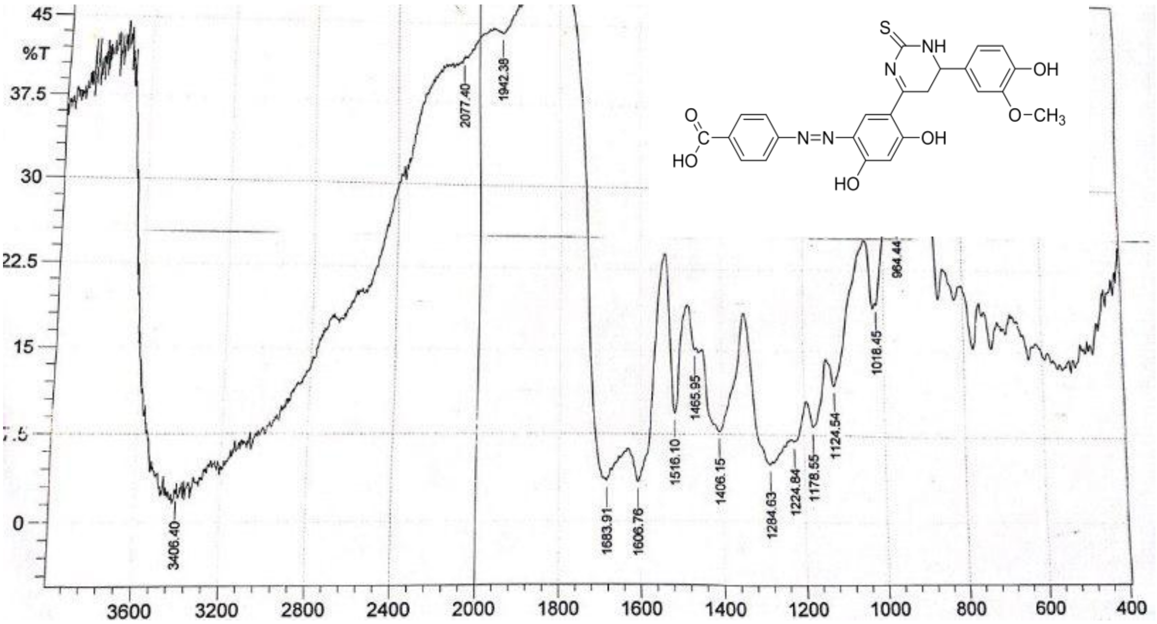
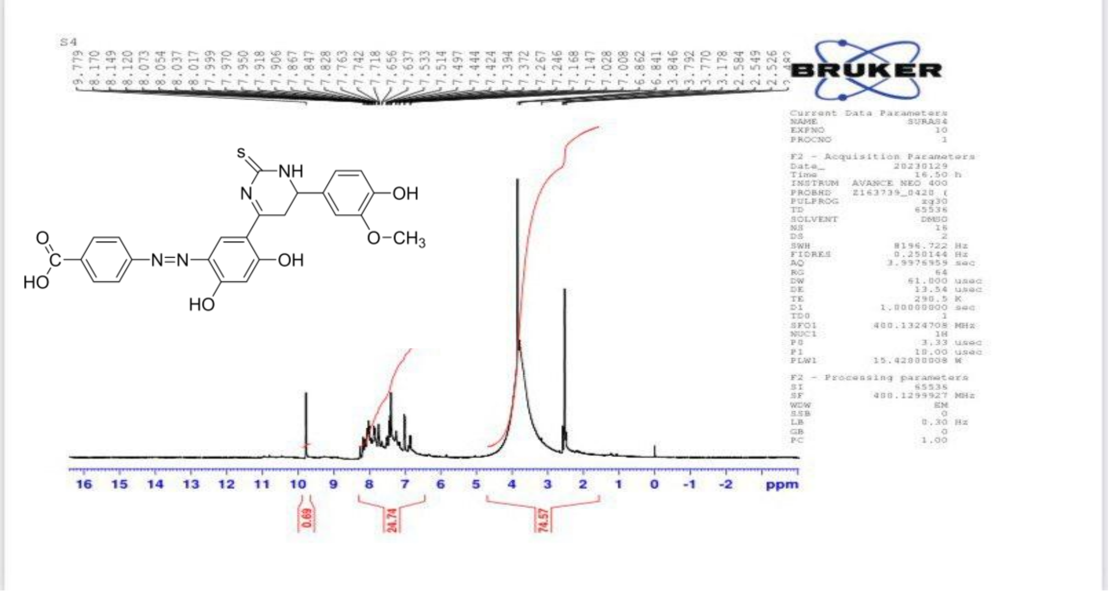


Figure (3-10) FT-IR Spectrum of compound[S4]



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Current Data Parameters
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PROCNO                 1

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PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ             3.9976959 sec
RG                    64
DW                61.000 usec
DE                 13.54 usec
TE                 298.5 K
D1            1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P0                  3.33 usec
P1                 10.00 usec
PLW1         15.42000008 W

F2 - Processing parameters
SI                 65536
SF           400.1299927 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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Figure (3-11) ^{1}H-NMR of compound [S4]

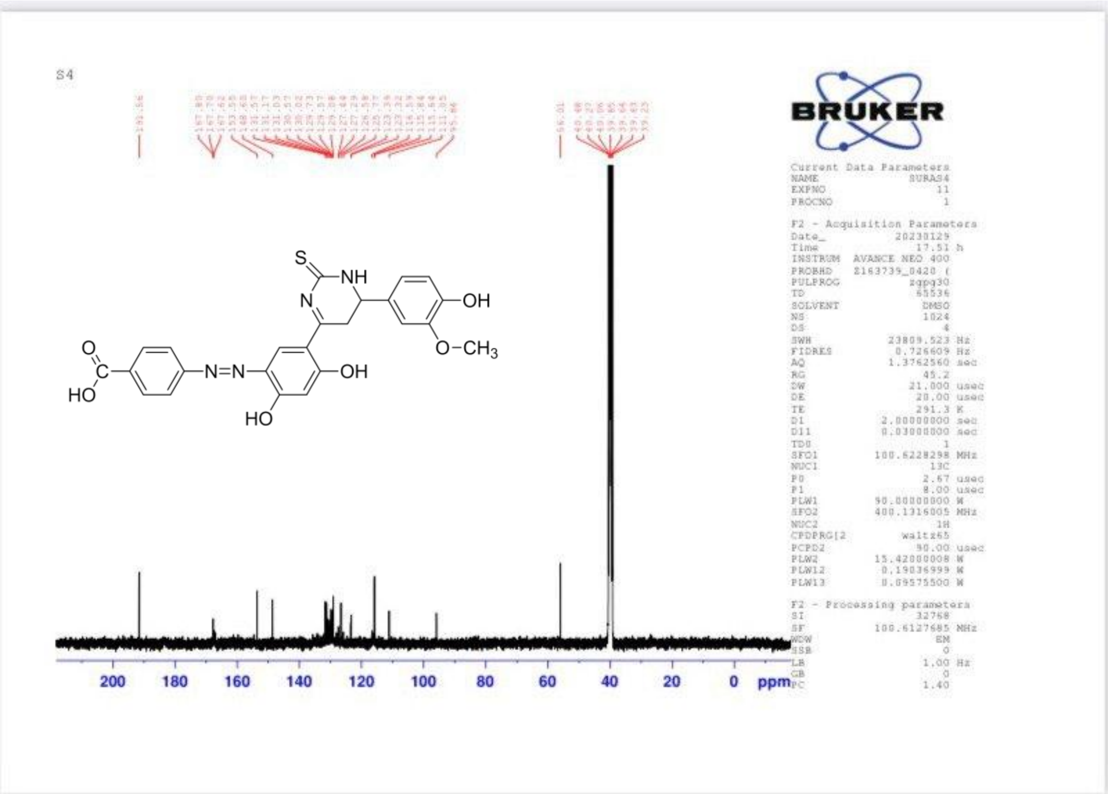


Figure (3-12) ^{13}C-NMR of compound [S4]

3.4 Synthesis of compounds[S5-S7]

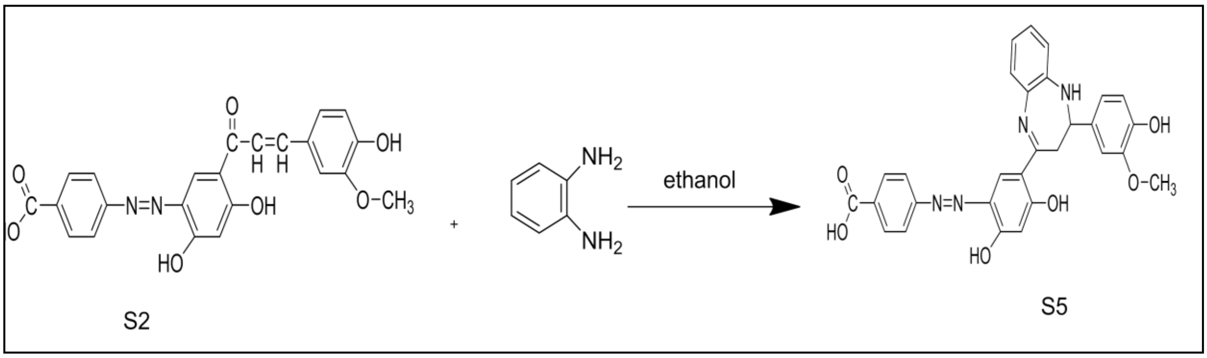
The chalcone[S2] reacted with few compounds(phenylindiamine,*o*aminophenol,*o*-aminothiophenol*)* to give new heterogeneous compounds in precence of absolute ethanol as a solvent,the compounds that prepare are seven member heterocyclic compounds[S5-S7],The FT-IR showed aband at (3437) for N-H in the FT-IR spectrum for [S5], while [S6,S7] don’t have band at that range ,and these compounds have a band at (1705,1650,1670) respectively for C=O$_{\text{carboxilic}}$.

The FTIR(*v*, cm^{-1}): spectrum of compound [S5] exhibited absorption band at (1278) cm-^{1} for O-CH$_{\text{ether}}$, (3173) for C-H $_{\text{Ar}}$,C=O $_{\text{carboxylic acid}}$ (1705), N-H (3437), N=N (1602).

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 5.8 for (H,OH) and signal at 6.3-7.8 for (10H, CH $_{\text{Arom}}$) ,3.887 for C-H$_{\text{alpha,}}$10.8 for (H,COOH).

.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at (148.7) for C-OH ,(154.1) for C,OR ,(172) for C,COOH, (41.3) for C,C-H $_{\text{endocyclic}}$.



Equation 3-4: Synthesis of Compound [S5]

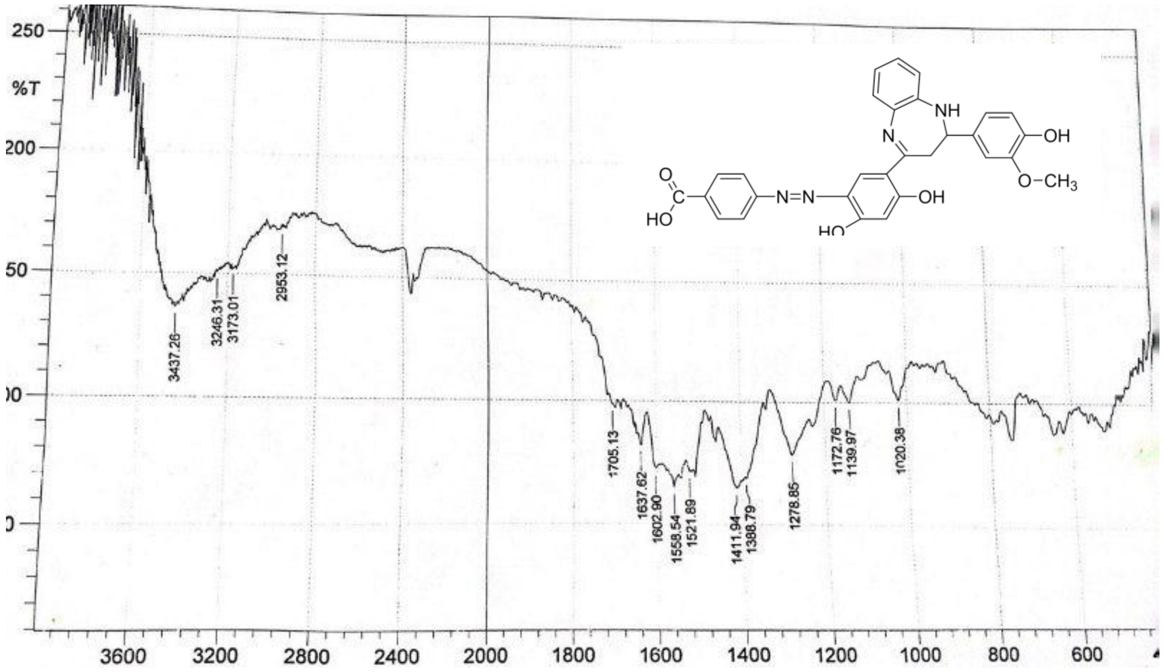


Figure (3-13) FT-IR Spectrum of compound[S5]

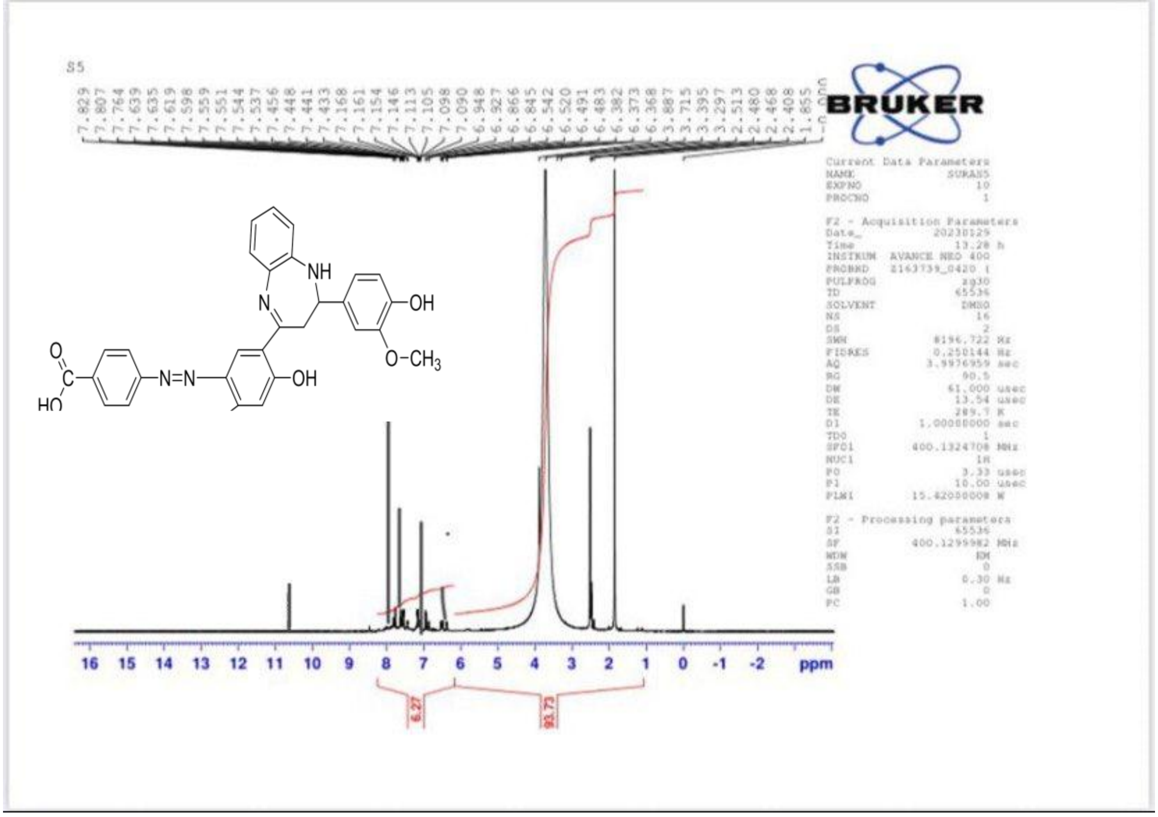


Figure (3-14) ^{1}H-NMR of compound [S5]

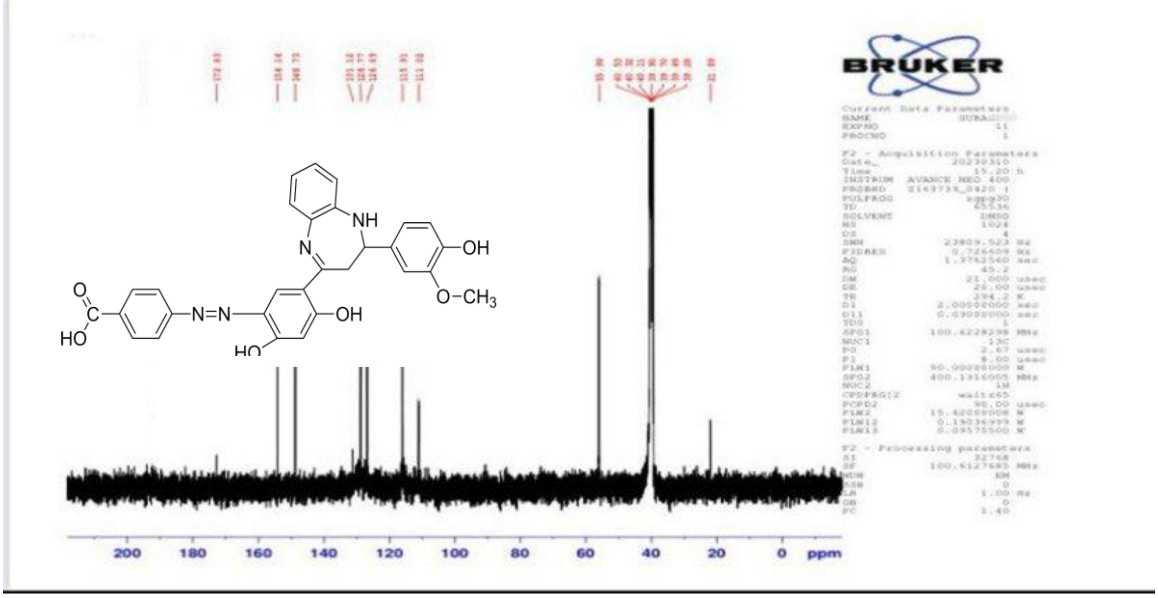
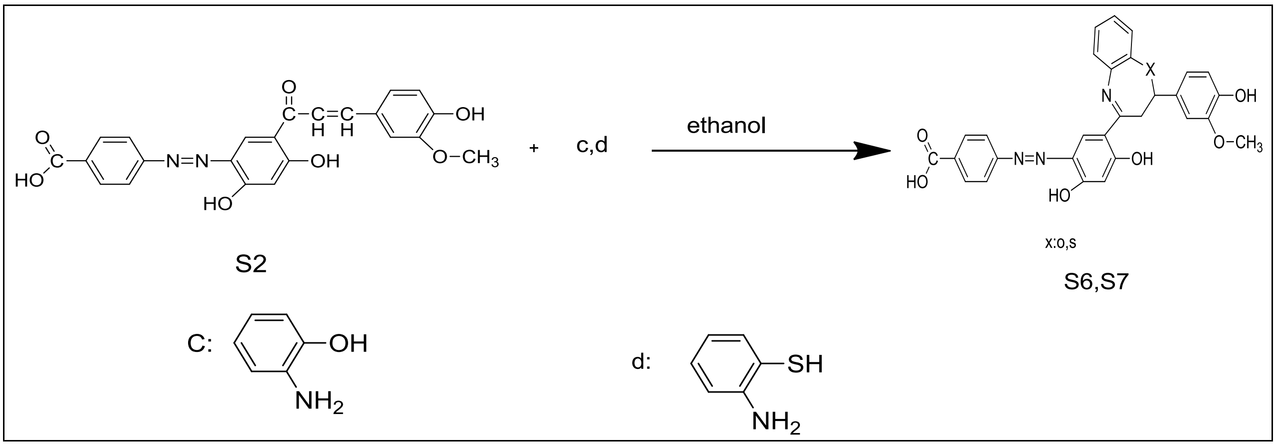


Figure (3-15) ^{13}C-NMR of compound [S5]



Equation 3-5: Synthesis of Compound [S6,S7]

The FTIR(v, cm^{-1}): spectrum of compound [S6] exhibited absorption band at (1280) cm-^{1} for O-CH$_{ether}$,C-H $_{Ar}$ (3173), C=O $_{carboxylic\ acid}$ (1650), N=N (1602), C=C$_{Arom}$ (1541).

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 5.8 for (H,OH) and signal at 6.3-7.8 for (12H, CH $_{Arom}$) ,3.846 for H,CH$_{alpha}$.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at (144.5) for C-OH ,(154.1) for C,OR , (115-145) for C,C-H$_{Arom}$,(41.1) for C,CH$_{Endocyclic}$

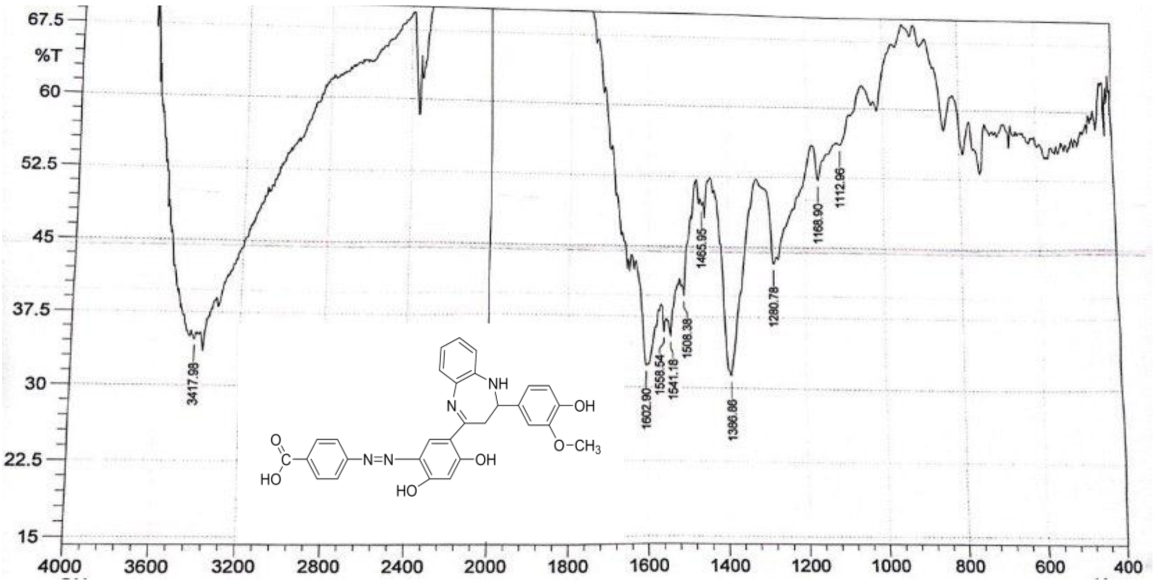
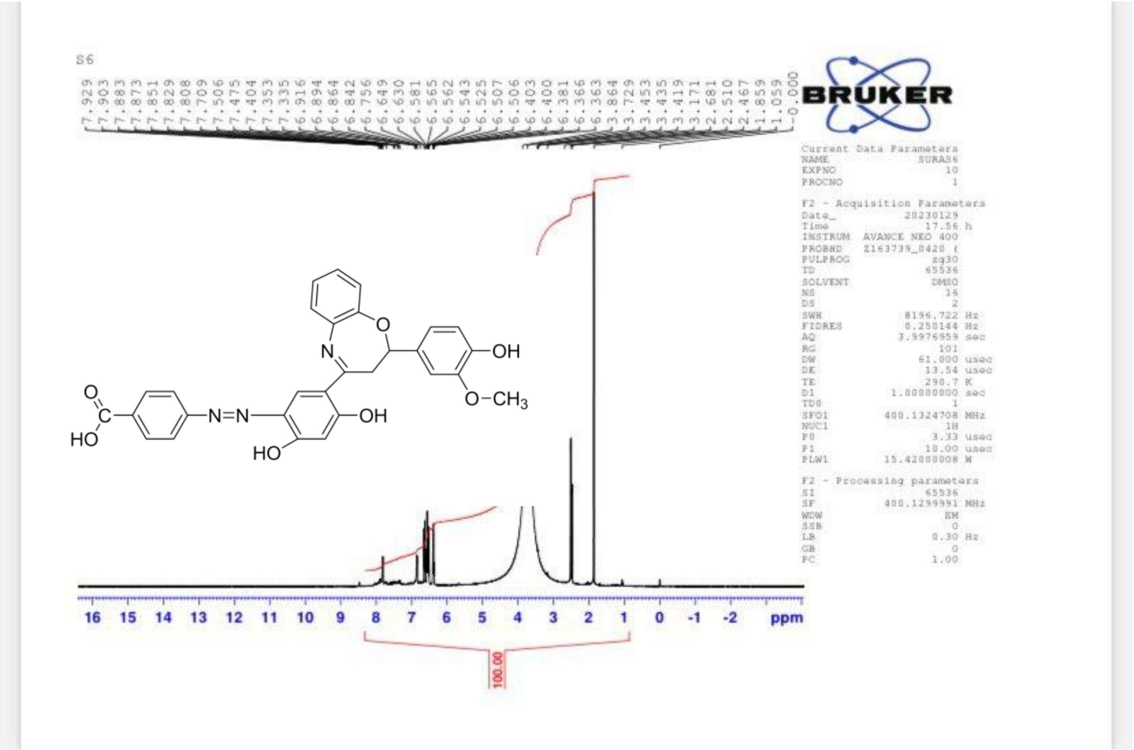
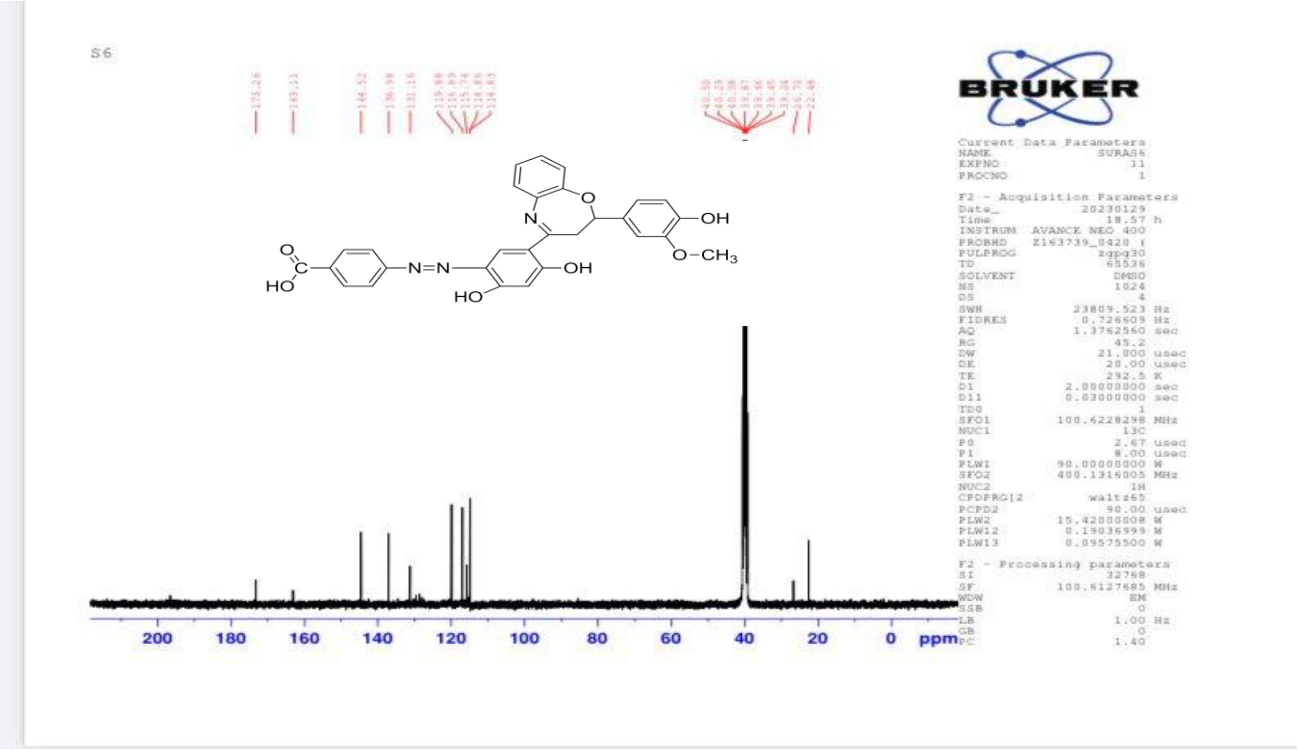


Figure (3-16) FT-IR Spectrum of compound[S6]



Figure(3-17) ^{1}HNMR of compound[S6]



Figure(3-18) ^{13}CNMR of compound[S6]

The FTIR (ν, cm^{-1}): spectrum of compound [S7] exhibited absorption band at (1650)for C=O $_{\text{carboxylic acid}}$ N=N (1602)and disappearance band at (1683) for C=O$_{\text{ketone}}$,C-H $_{\text{Ar}}$ (3080).

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 5.47 for (H,OH) and signal at 6.4-7.8 for (12H, CH $_{\text{Arom}}$) , (C-H$_{\text{Alpha}}$):3.81, (C-H$_{\text{Endocyclic}}$):1.8

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at (155.6) for C-OH ,(56.02) for C-H$_{\text{Alpha}}$, (115-135) for C,C-H $_{\text{Arom}}$, (40.1) for C,C-H$_{\text{Endocyclic}}$.

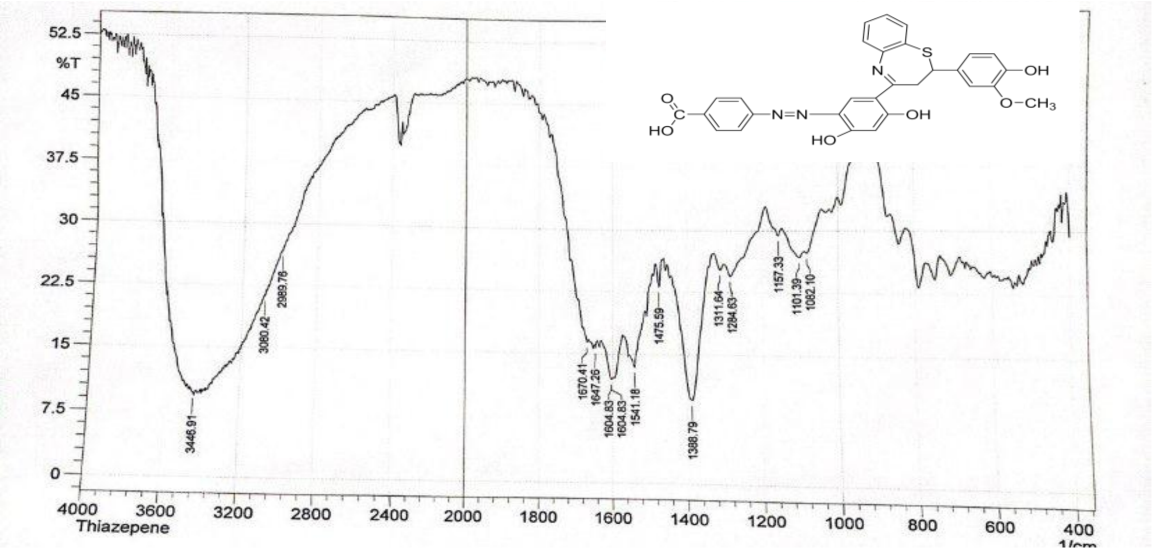
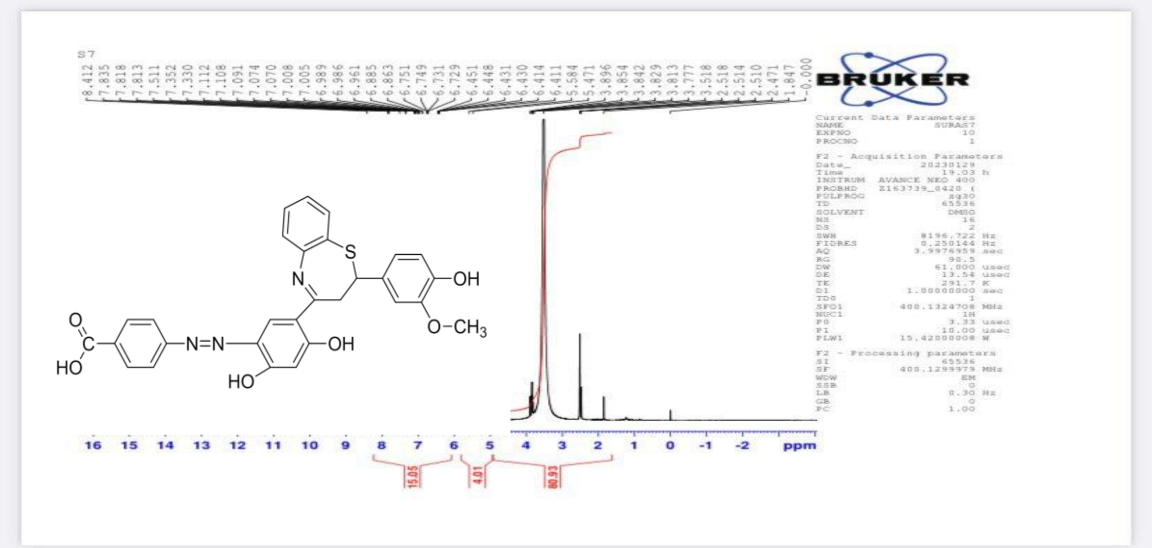


Figure (3-19) FT-IR Spectrum of compound[S7]



Figure(3-20) ^{1}HNMR of compound[S7]

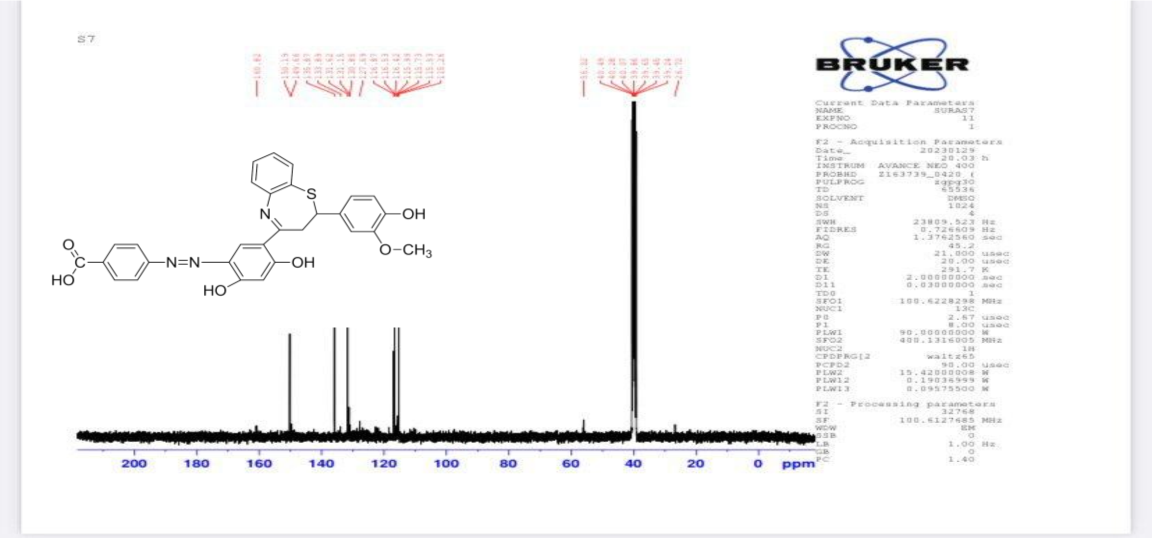


Figure (3-21) ^{13}C-NMR of compound [S7]

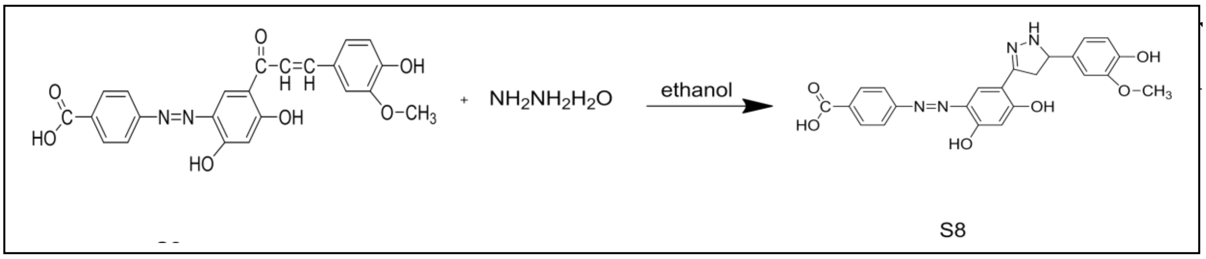
3.5 Synthesis of compound [S8,S9]

(Pyrazol S5, Isoxazol S6) These compounds with a five-ring were prepared from the interaction of (hydrazine hydrate, hydroxylamine hydrochloride) compounds with ethanol in the presence of a few drops of sodium hydroxide with the presence of a reflex reaction. Paraffin oil was used during the reflex process so that the heat is distributed evenly on all parts of the round flask. These two reactions formed five heterogeneous cyclic compounds. Through the experiment, it turned out that the compound isoxazole[S9] is a somewhat viscous compound, so there was difficulty in dealing with it.

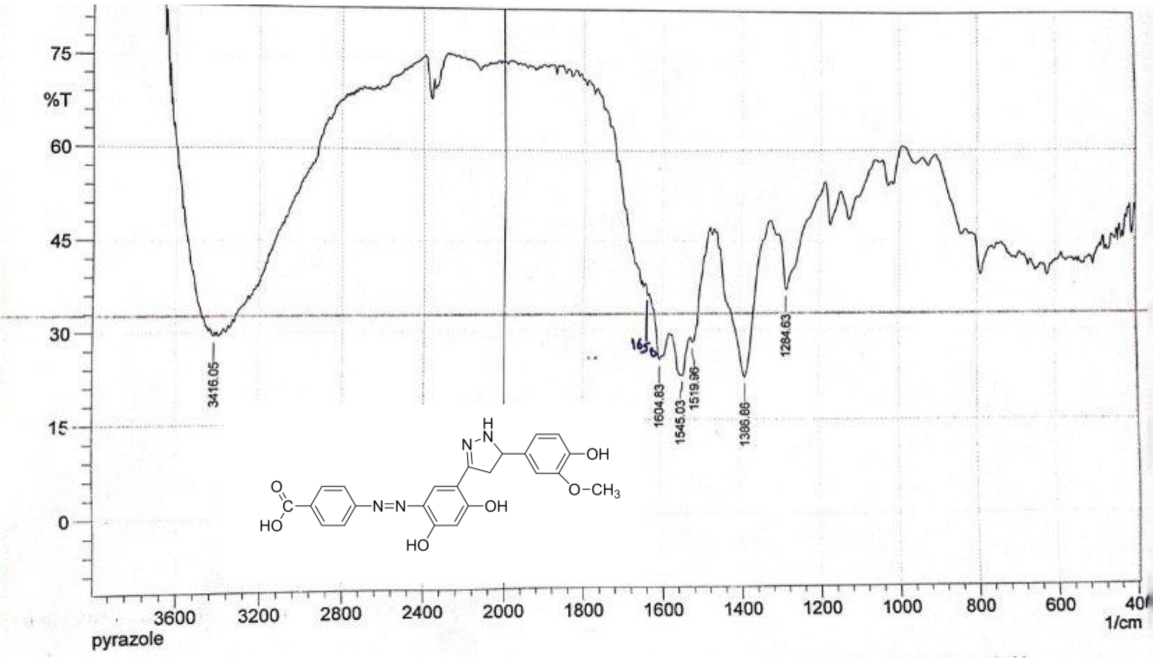
The FT-IR (*v*, cm^{-1}): spectrum of compound [S8] show some band:C-H $_{Ar}$ (3080), C=O $_{carboxylic\ acid}$ (1650), N-H (3416), N=N (1604).

^{1}H-NMR (400MHz,DMSO-d6) :(δ, ppm) (7H,Ar-H) : 6.4-8.3,C-H$_{Alpha}$ (3.81) ,O-H:6.44,C-H $_{Endocyclic}$:1.9

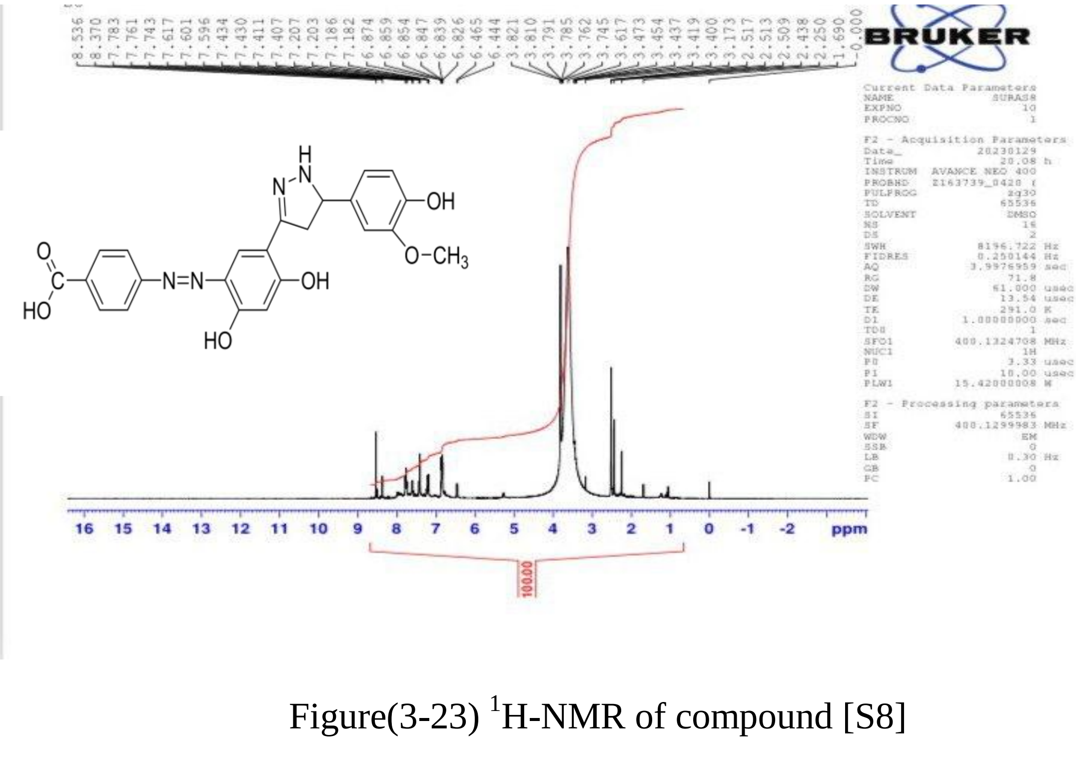
^{13}C-NMR (100MHz,DMSO-d6) :(δ, ppm): (110-131) for (18C,Ar-C),(169.30) for C=O$_{carboxylic\ acid}$ C,OH : 148.8 , (55.88) :C-H$_{Alpha}$,(40,51) :C,C-H $_{Endocyclic}$.



Equation 3-6: Synthesis of Compound [S8]



Figure(3-22) FT-IR Spectrum of compound [S8]



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PROCNO                 1

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PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ             3.9976959 sec
RG                  71.8
DW                61.000 usec
DE                 13.54 usec
TE                 291.0 K
D1            1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P0                  3.33 usec
P1                 10.00 usec
PLW1         15.42000008 W

F2 - Processing parameters
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SF           400.1299983 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
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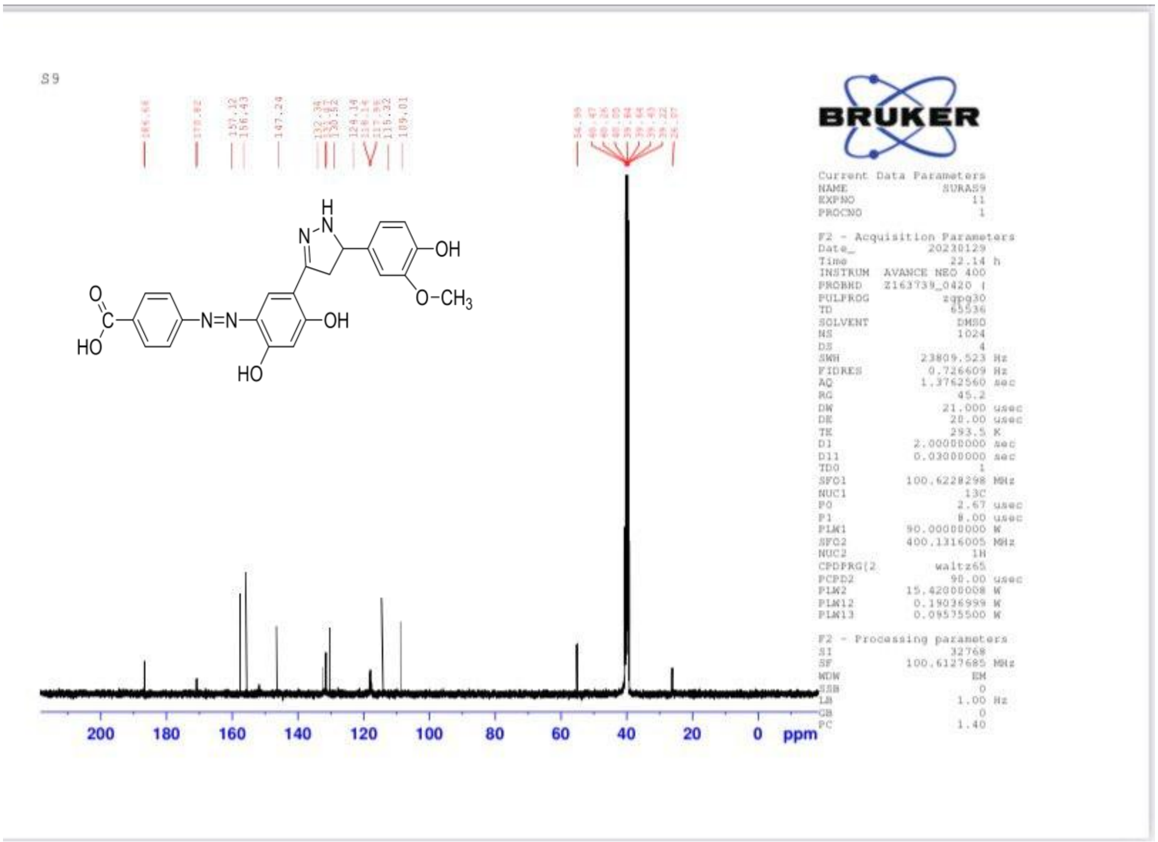
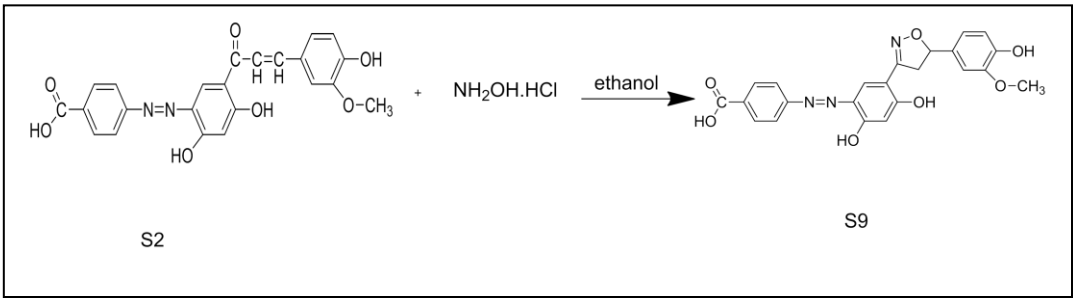
Figure(3-23) ^{1}H-NMR of compound [S8]



Figure(3-24) ^{13}C-NMR of compound [S8]



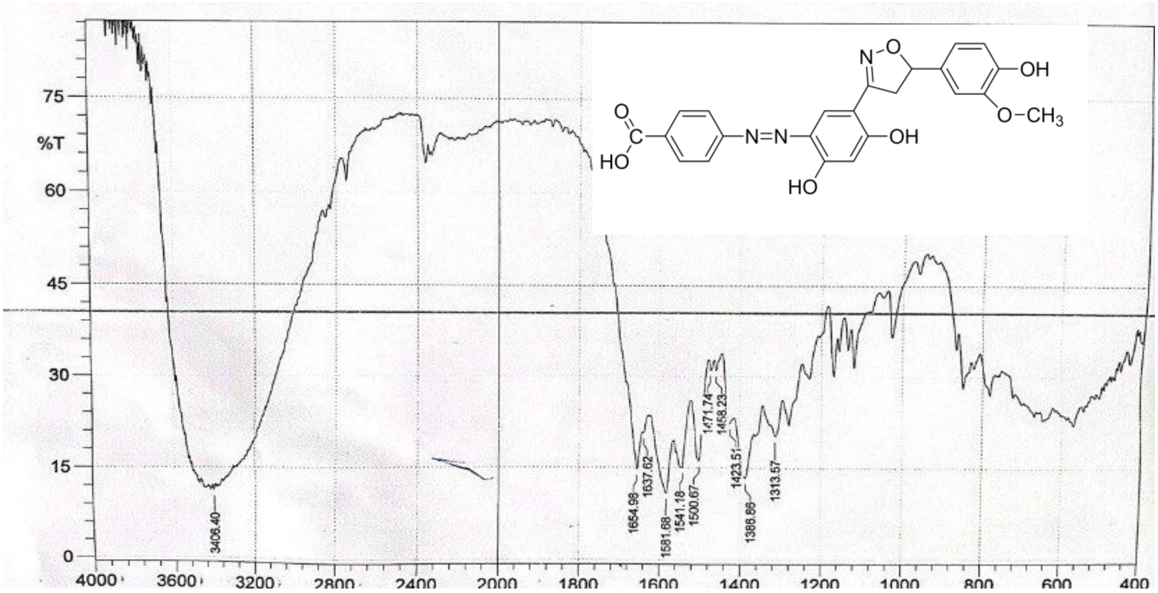
Equation 3-7: Synthesis of Compound [S9]

The FT-IR(v, cm^{-1}): spectrum of compound [S9] show some band as show in below:

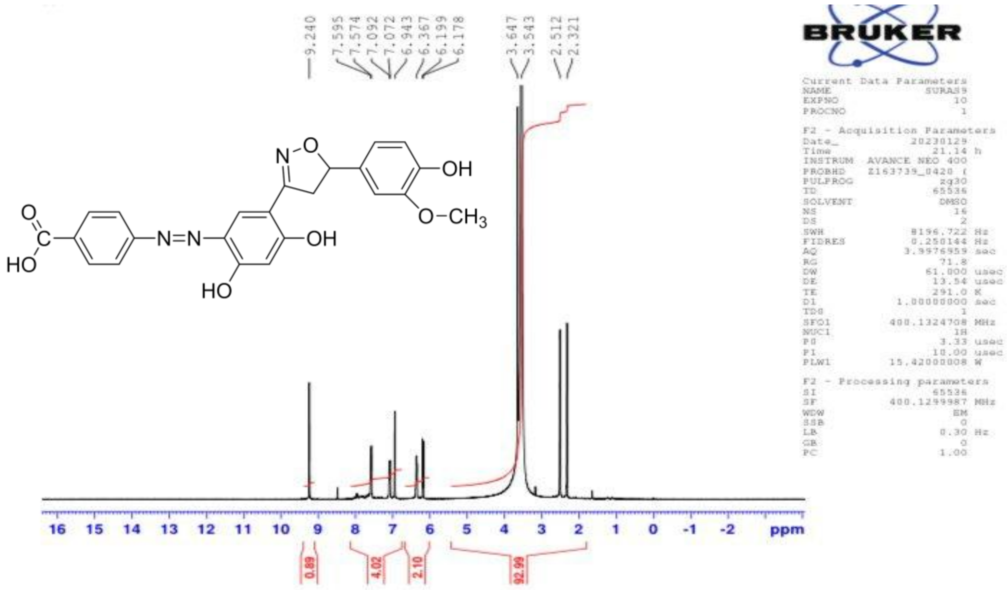
C-H $_{Ar}$ (3100), C=O $_{carboxylic\ acid}$ (1654), N=N (1581). And disapperence band at (1683) for C=O$_{Keton}$.

^{1}H-NMR (400MHz,DMSO-d6) :(δ, ppm) (7H,Ar-H) : 6.1-7.5, C-H $_{Alpha}$ (3.6) ,O-H : 9.2 ,C-H$_{Endocyclic}$:1.7

^{13}C-NMR (100MHz,DMSO-d6) :(δ, ppm) (117-131) for (12C,Ar-C),(170.8) for C=O$_{\text{carboxylic acid}}$, (54.3) C,C-H $_{\text{Alpha}}$,(40.4) C,C-H$_{\text{Endocyclic}}$.



Figure(3-25) FT-IR Spectrum of compound [S9]



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PROCNO                 1

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PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ             3.9976959 sec
RG                  71.8
DW                61.000 usec
DE                 13.54 usec
TE                 291.0 K
D1            1.00000000 sec
TD0                    1
SFO1         400.1324708 MHz
NUC1                  1H
P0                  3.33 usec
P1                 10.00 usec
PLW1         15.42000008 W

F2 - Processing parameters
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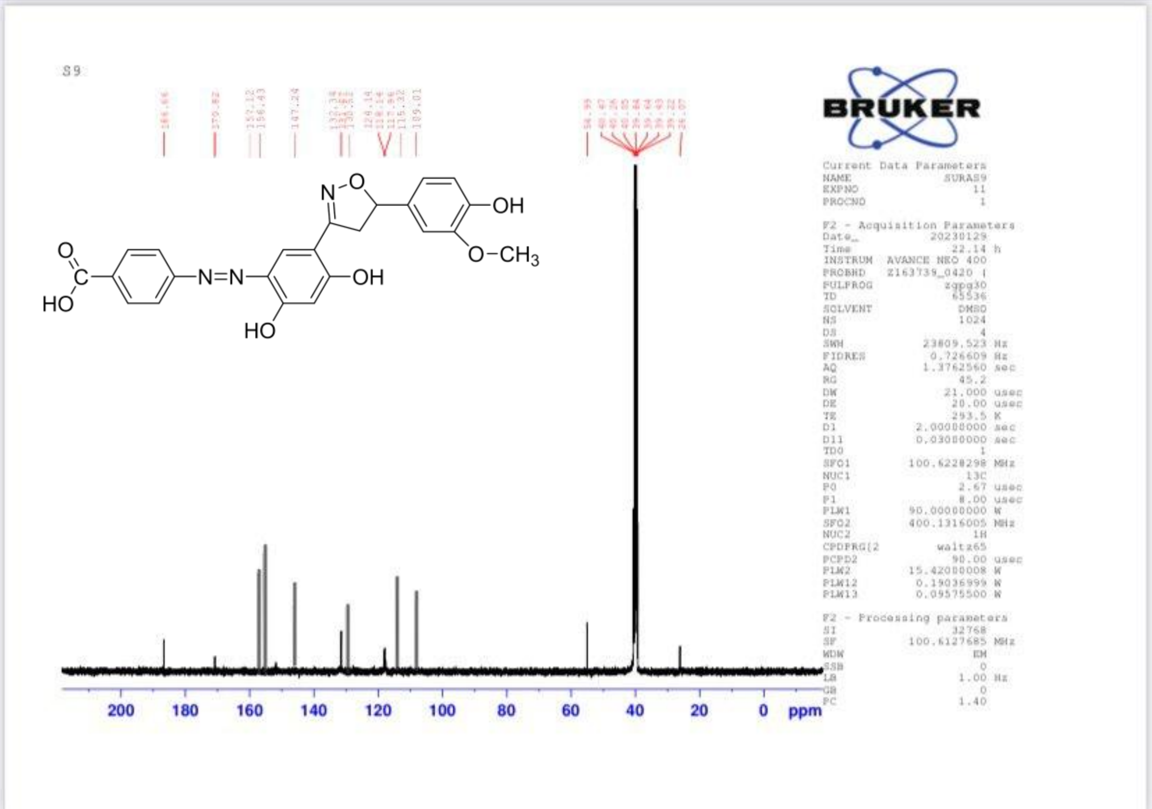
Figure(3-26) ^{1}H-NMR of compound [S9]



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PROCNO                 1

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PULPROG           zgpg30
TD                 65536
SOLVENT             DMSO
NS                  1024
DS                     4
SWH            23809.523 Hz
FIDRES          0.726609 Hz
AQ             1.3762560 sec
RG                  45.2
DW                21.000 usec
DE                 20.00 usec
TE                 293.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1
SFO1         100.6228298 MHz
NUC1                 13C
P0                  2.67 usec
P1                  8.00 usec
PLW1         90.00000000 W
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz65
PCPD2              90.00 usec
PLW2         15.42000008 W
PLW12         0.19036999 W
PLW13         0.09575500 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

Figure(3-27) ^{13}C-NMR of compound [S9]

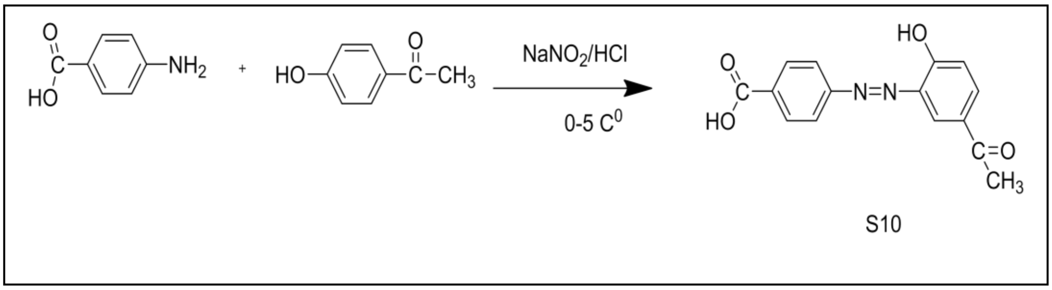
3.6. Synthesis of compound [S10]

The first reaction involve reaction of *p*-aminobenzoic acid as nucleophile (rich in electron) with nitrous ion to form diazonium salt, after (15 min) this diazonium reaction with coupling compound (4- hydroxylbenzaldehyd), The reaction should be carried out at (0-5°C) with continuous stirring to form azo dye.

The FTIR(*v*, cm^{-1}): spectrum exhibited absorption band at (1585) cm^{-1} for N=N and disappearance absorption band at (3363,3473) cm^{-1} for $NH_{2,}$ O-H (3267),C-H $_{\text{Ar}}$ (3101), C=O $_{\text{carboxylic acid}}$ (1654), C=O$_{\text{ketone}}$ (1660).

^{1}HNMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 7.5-8.2 for (6H,Ar-H),5.9 for H-OH, 2.5 for C-H $_{\text{Alpha}}$.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at 119-132.4 for (12C,Ar C), 196.6 for C=O$_{\text{keton}}$,167.7 C=O $_{\text{carboxylic}}$, 154 C- OH, 26 C-H$_{\text{Alpha}}$



Equation 3-8: Synthesis of Compound [S10]

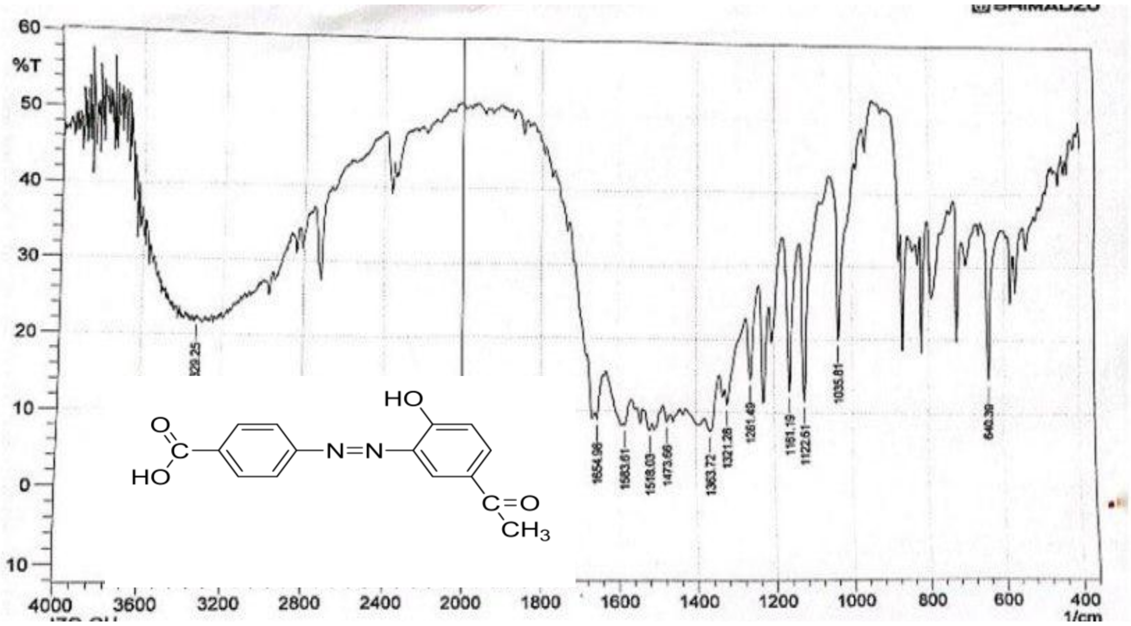
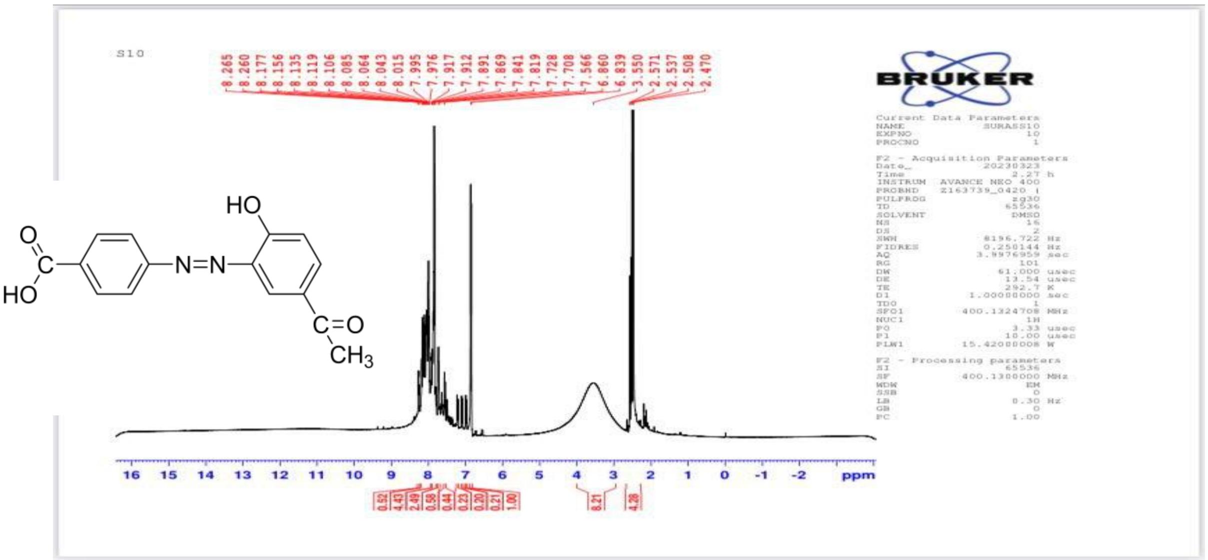
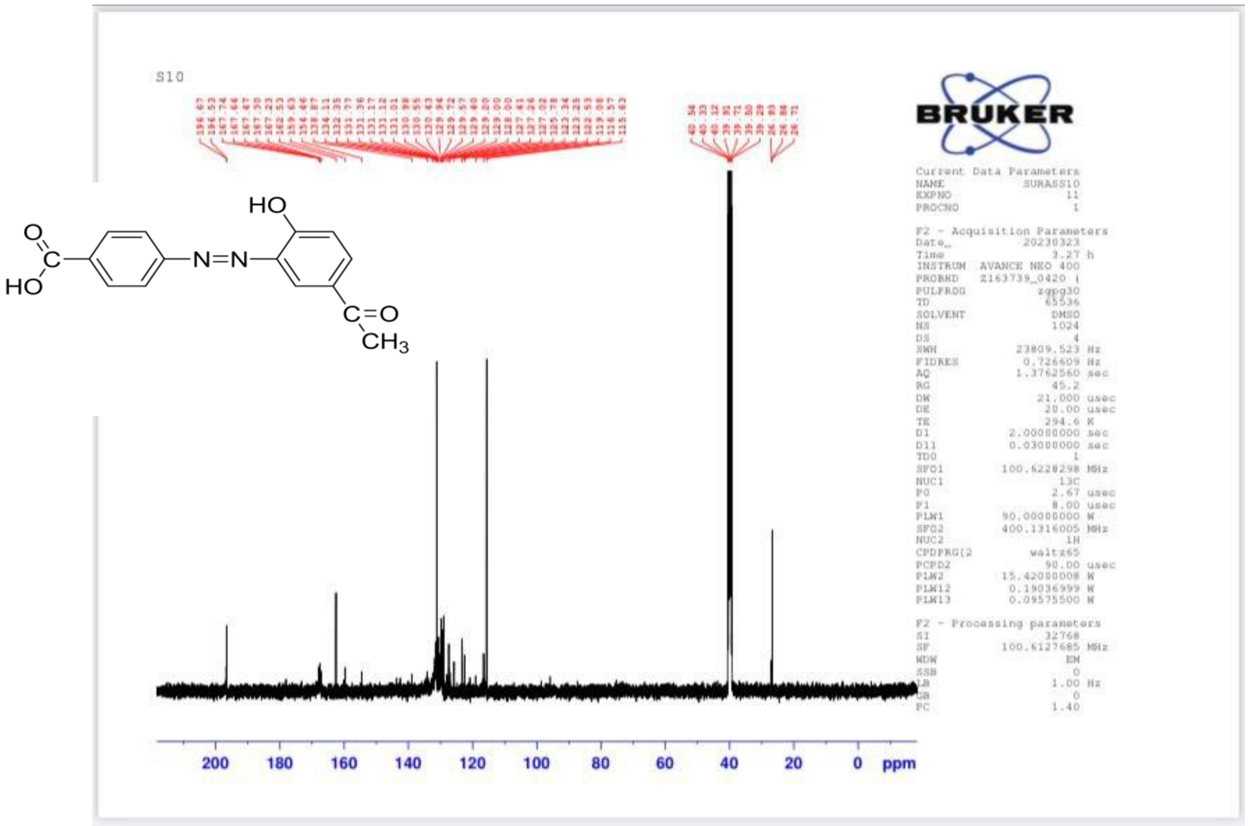


Figure (3-28) FT-IR Spectrum of compound[S10]



Figure(3-29) ^{1}H-NMR of compound [S10]



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PROCNO                   1

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PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                   1024
DS                      4
SWH             23809.523 Hz
FIDRES           0.726609 Hz
AQ              1.3762560 sec
RG                   45.2
DW                 21.000 usec
DE                  20.00 usec
TE                  294.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1          100.6228298 MHz
NUC1                  13C
P0                   2.67 usec
P1                   8.00 usec
PLW1          90.00000000 W
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz65
PCPD2               90.00 usec
PLW2          15.42000008 W
PLW12          0.19036999 W
PLW13          0.09575500 W

F2 - Processing parameters
SI                  32768
SF            100.6127685 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
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Figure(3-30) ^{13}C-NMR of compound [S10]

3.7. Synthesis of compounds[S11]

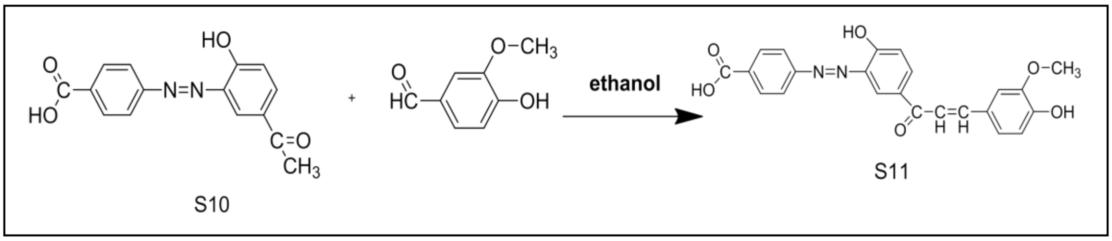
Chalcone [S11] synthesized from azo dye(4-((5-acetyl-2,4-dihydroxyphenyl)diazenyl) benzoic acid [S10] with aldehyde such as vanillin(4-hydroxy benzaldehyd) in presence of ethanol as solvent and (10% NaOH ,dissolved in mix of(ethanol and distill water) as catalyst, When the hydroxyl group in the base accepts the hydrogen atom in the carbonyl group's alpha position, an aldol condensation process takes place, resulting in the release of a water molecule , it was found that the absorbance was shifted due to the entry of a withdrawn group, which is the ether group (C- OR), in an aldehyde to synthesis chalcone.

FT-IR (v, cm^{-1}): spectrum show band at 1654 for C=O$_{\text{carboxylic}}$, (1604) cm^{-1} for N=N, O-H (3267),C-H $_{\text{Ar}}$ (3101), C=O$_{\text{ketone}}$ (1660) .

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 6.1-7.5 for (6H,CH$_{\text{Arom}}$) and signal at 3.6 for (H,CH$_{\text{Alpha}}$) ,(C-H$_{\text{Endocyclic}}$) :1.65 .

.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at 110-140 for (18C,Ar-C)and signal at 26.6 for,C-H_{alpha} , (165) for C=$O_{carboxylic\ acid}$ (180) for C=O_{ketone}



Equation 3-9: Synthesis of Compound [S11]

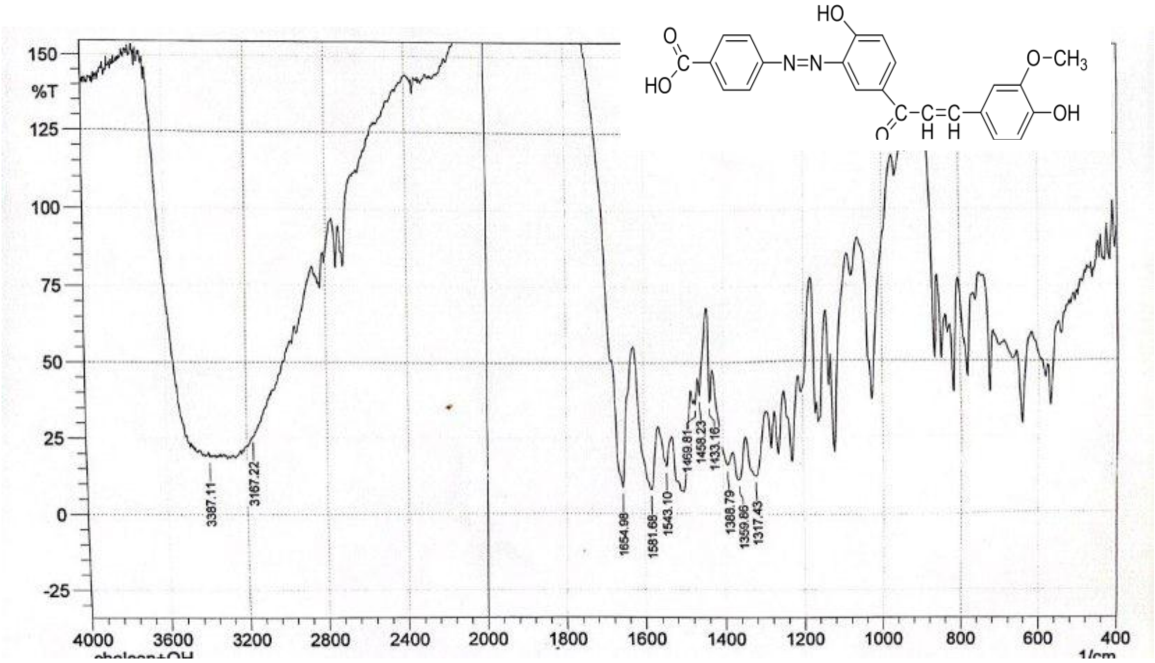
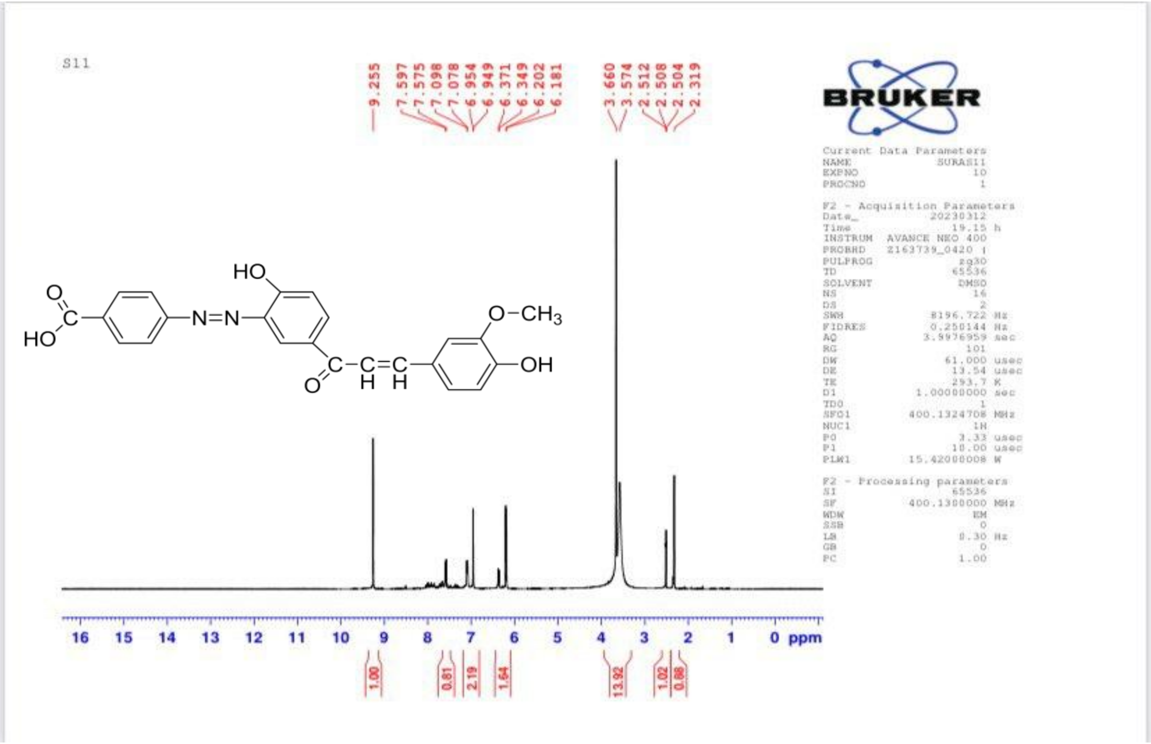


Figure (3-31) FT-IR Spectrum of compound[S11]



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PROCNO                  1

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SOLVENT              DMSO
NS                     16
DS                      2
SWH              8196.722 Hz
FIDRES           0.250144 Hz
AQ              3.9976959 sec
RG                    101
DW                 61.000 usec
DE                  13.54 usec
TE                  293.7 K
D1             1.00000000 sec
TD0                     1
SFO1          400.1324708 MHz
NUC1                   1H
P0                   3.33 usec
P1                  10.00 usec
PLW1          15.42000008 W

F2 - Processing parameters
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PC                   1.00
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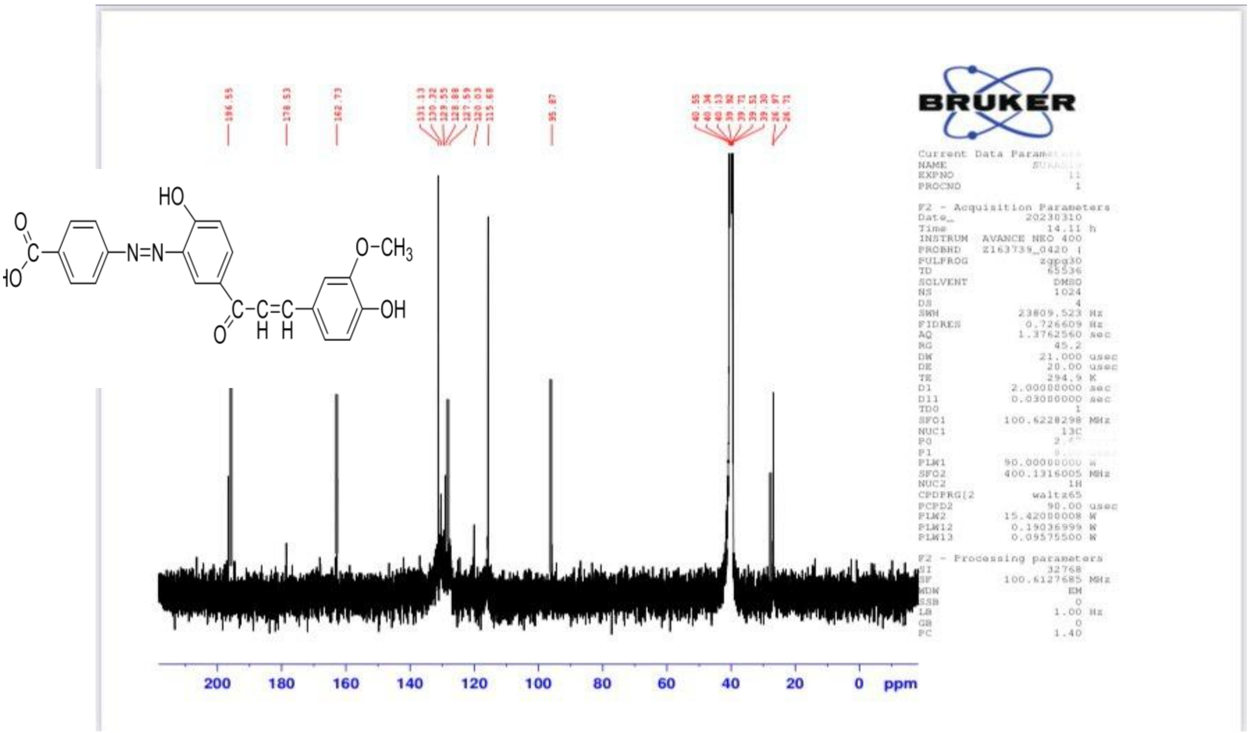
Figure (3-31) ^{1}H NMR of compound[S11]



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TD                  65536
SOLVENT              DMSO
NS                   1024
DS                      4
SWH             23809.523 Hz
FIDRES           0.726609 Hz
AQ              1.3762560 sec
RG                   45.2
DW                 21.000 usec
DE                  20.00 usec
TE                  294.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1          100.6228298 MHz
NUC1                  13C
P0             [illegible]
P1             [illegible]
PLW1          90.00000000 W
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz65
PCPD2               90.00 usec
PLW2          15.42000008 W
PLW12          0.19036999 W
PLW13          0.09575500 W

F2 - Processing parameters
SI                  32768
SF            100.6127685 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

Figure(3-32) ^{13}C-NMR of compound [S11]

3.10 Synthesis of compound[S12,S13]:

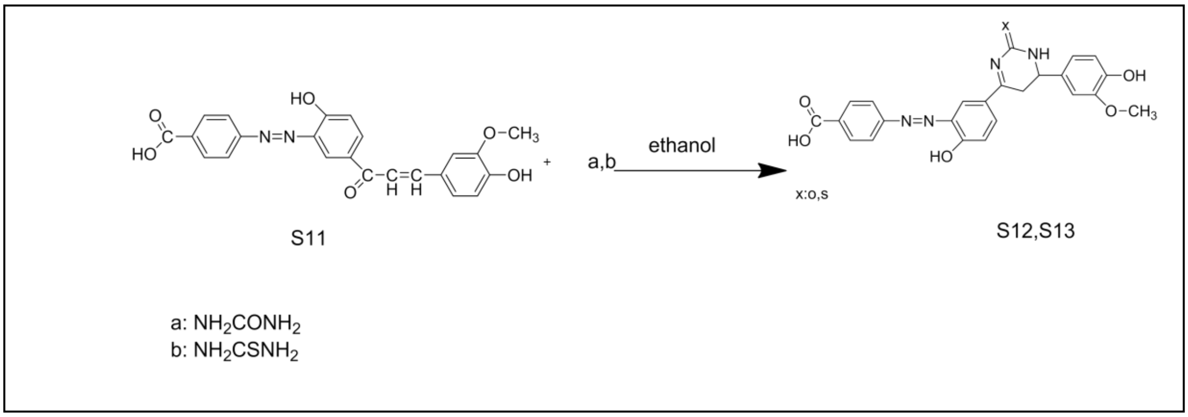
(Oxazine S12,Thiazen S13) These compounds were prepared from the reaction of (urea and thiourea) with ethanol in the presence of a few drops of sodium hydroxide with continuous stirring in an ice bath, and heterocyclic compounds with a hexagonal ring were produced when the chalcone interacted with urea as a result of that a viscous compound, while a powdery compound was produced when it interacted with thiourea. After the amine group disappeared in the first reaction, it reappeared in compounds [S12,S13] (3481,3483) as a result of the interaction of the chalcone with urea and thiourea.

The FTIR(v, cm^{-1}): spectrum of compound [S12] exhibited absorption band at (1581) cm^{-1} for N=N and (1654) cm^{-1} for $C=O_{carboxylic}$,(1228) cm^{-1} for C,OR_{Ether} O-H (3338), C-H Ar (3063), $C\text{-}H_{alph}$ (2829).

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 5.4 for (H,OH) and signal at 3.6 for (H,CH_{alpha}), (6H,Ar-H) : 6.85-8.08 ,(H-NH) :8.5.

.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at 117-131for (12C,Ar-C)and signal at 54.99 for,$C\text{-}H_{alpha}$, (167.26) for $C=O_{carboxylic\ acid}$ (196.57) for $C=O_{ketone}$, (40.5) for $C,CH_{Endocyclic}$.



Equation 3-10: Synthesis of Compound [S12,S13]

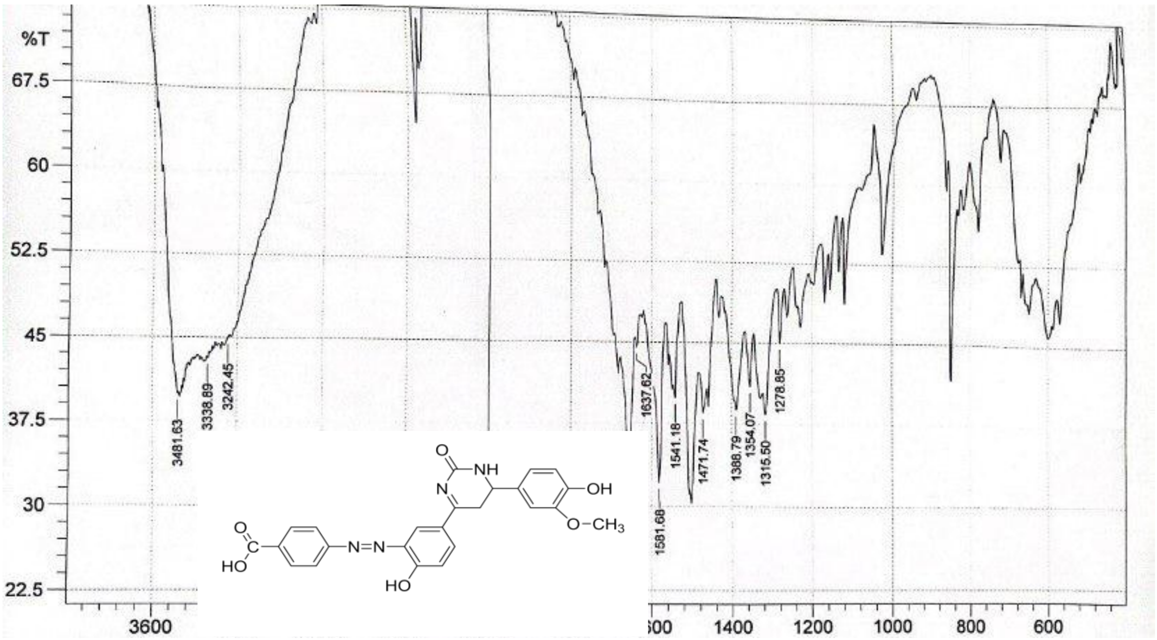


Figure (3-33) FT-IR Spectrum of compound [S12]

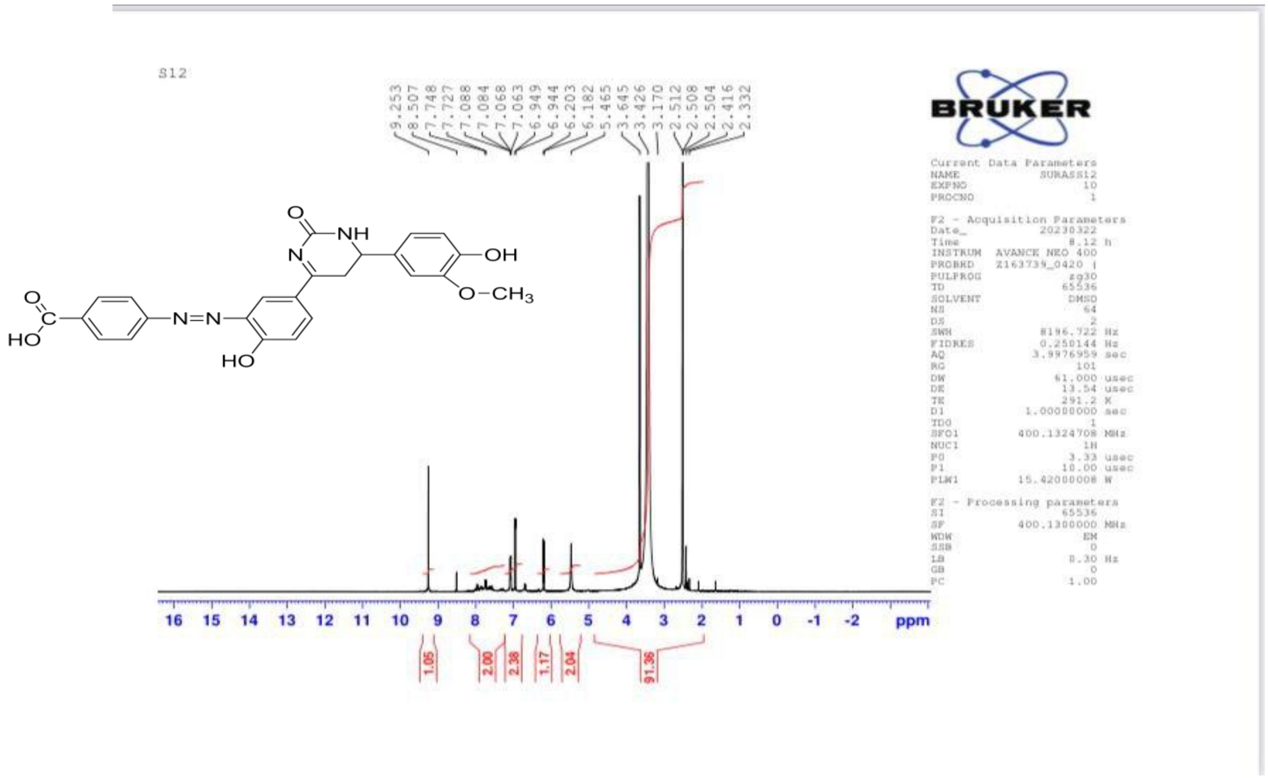
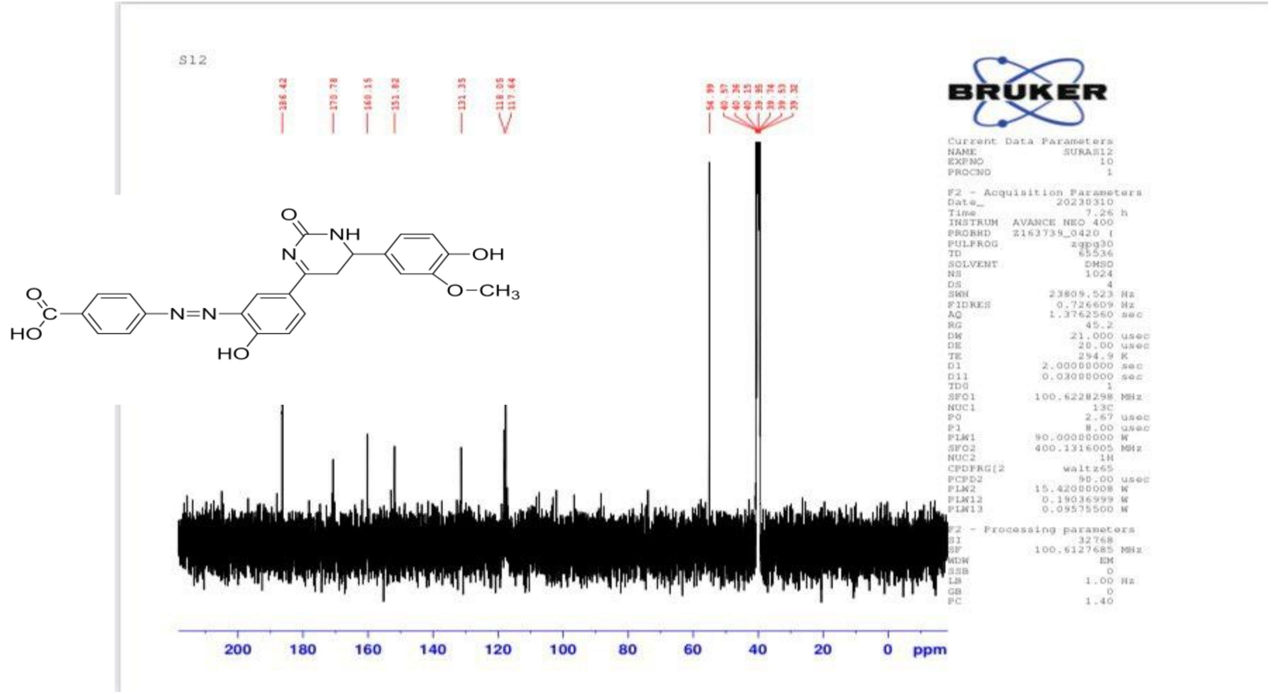


Figure (3-34) ^{1}H NMR of compound[S12]



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PROCNO                  1

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PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                   1024
DS                      4
SWH             23809.523 Hz
FIDRES           0.726609 Hz
AQ              1.3762560 sec
RG                   45.2
DW                 21.000 usec
DE                  20.00 usec
TE                  294.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1          100.6228298 MHz
NUC1                  13C
P0                   2.67 usec
P1                   8.00 usec
PLW1          90.00000000 W
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz65
PCPD2               90.00 usec
PLW2          15.42000008 W
PLW12          0.19036999 W
PLW13          0.09575500 W

F2 - Processing parameters
SI                  32768
SF            100.6127685 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

Figure (3-35) ^{13}CNMR of compound[S12]

The FTIR spectrum of compound [S13] exhibited absorption band at (1581) cm^{-1} for N=N and (3483) cm^{-1} for N-H.
IR (*v*, cm^{-1}): O-H (3267),C-H Ar (3101), C=O $_{\text{carboxylic acid}}$ (1691), C=S : (1654) , N-H: (3200).

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 5.49 for (H,OH) and signal at 1.9 for (H, CH $_{\text{Endocyclic}}$), (10H,Ar-H) : 6.12-7.6 .
.
^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at 95-132for (18C,Ar-C), and signal at 55.72 for, C-H $_{\text{alpha}}$, (170.8) : C=O $_{\text{carboxylic acid}}$

(40.7) : C,C-H $_{\text{Endocyclic}}$ (186) :C,C-N $_{\text{Endocyclic}}$.

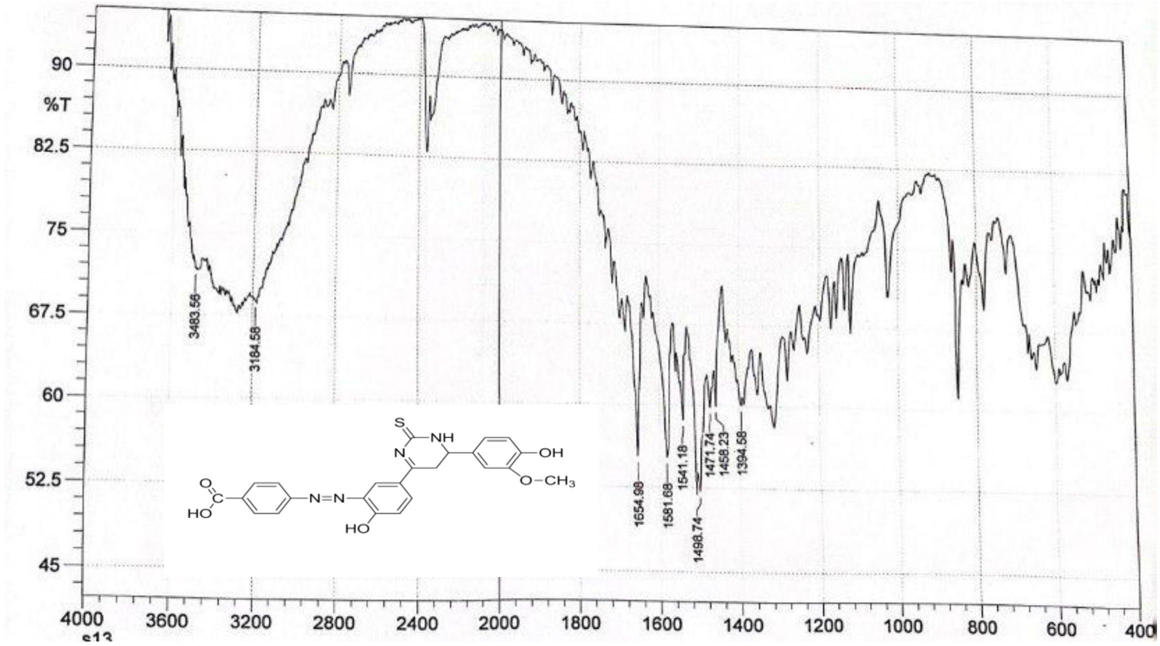


Figure (3-36) FT-IR Spectrum of compound[S13]

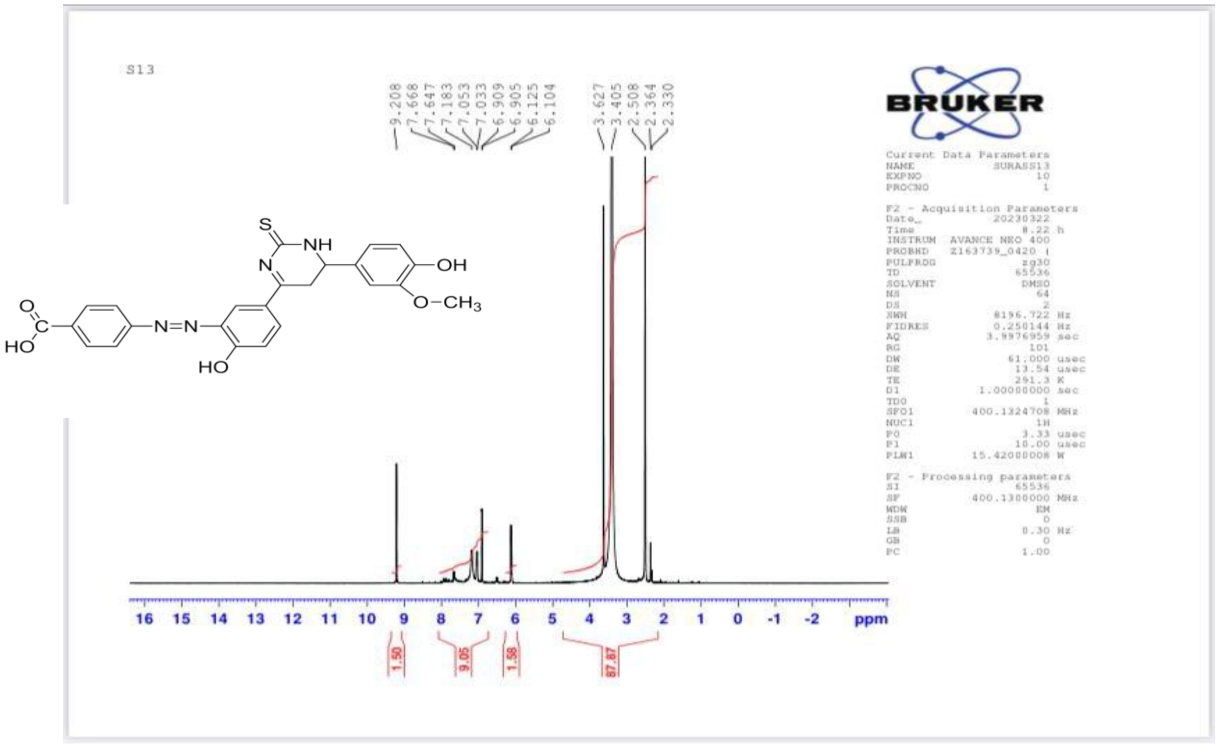


Figure (3-37) ^{1}H NMR of compound[S13]

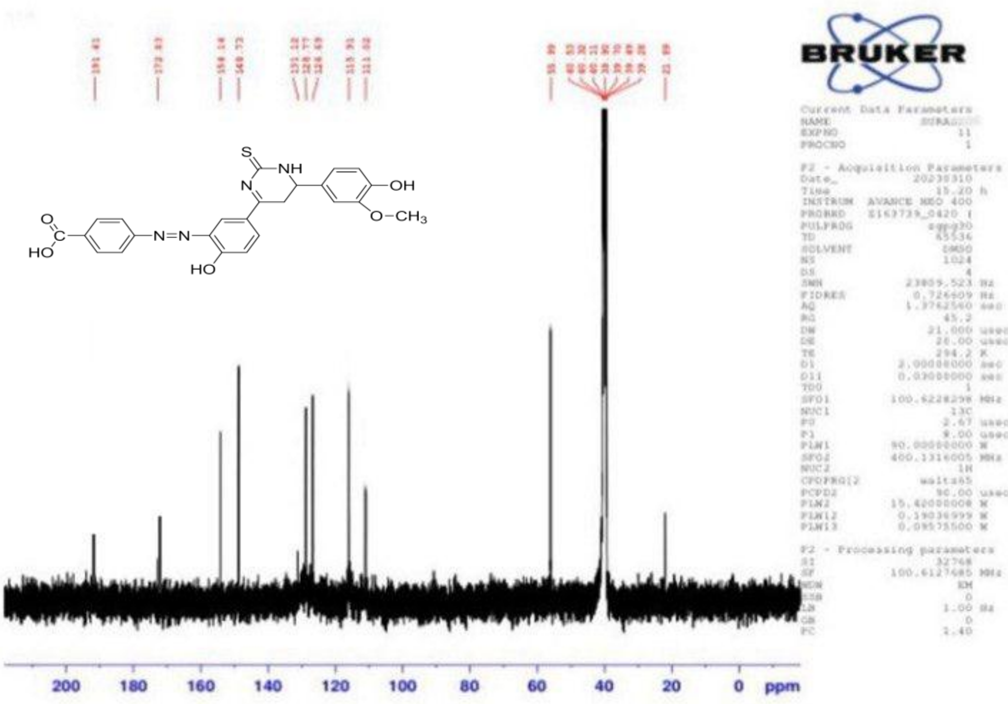


Figure (3-38) ^{13} CNMR of compound[S13]

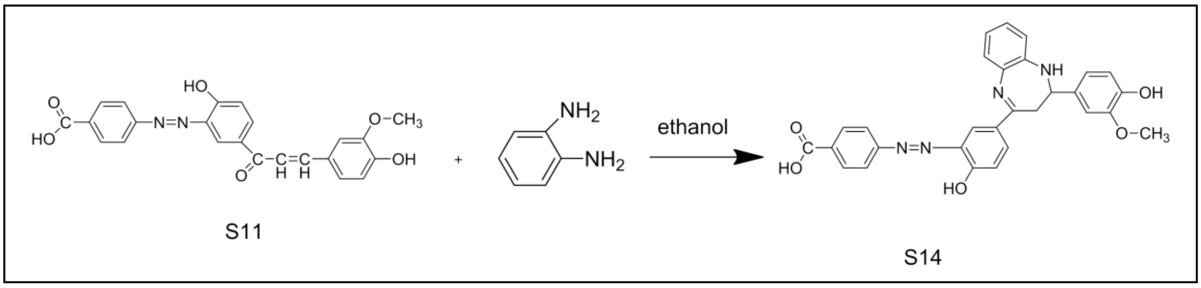
3.11 Synthesis of compounds[S14-S16]

The chalcone[S11] reacted with some compounds(phenylin diamine,*o*-amino phenol,*o-aminothiophenol)* to give new seven heterogeneous compounds in precence of absolute ethanol as a solvent,the compounds that prepare are seven member heterocyclic compounds[S14-S16],The FT-IR showed aband at (3406) for N-H in the FT-IR spectrum for [S14]

FT-IR(*v*, cm^{-1}) spectrum of compound [S14] exhibited absorption band at (1278) $cm-^{1}$ for O-CH_{ether} ,(3177) for C-H_{Ar} ,C=$O_{carboxylic\ acid}$ (1650), N-H (3406) .

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 5.49 for (H,OH) and signal at 6.7-8.5 for (18H, CH $_{Arom}$) , (HCOOH) :11.1, (N-H) :3.82, (C-$H_{Endocyclic}$) :1.9.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at (148.3) for C-OH ,(56.5) for C-H_{Alpha} , (110-152.4) : (18C,Ar-C) , (39.36) : C,C-$H_{Endocyclic}$



Equation 3-11: Synthesis of Compound [S14]

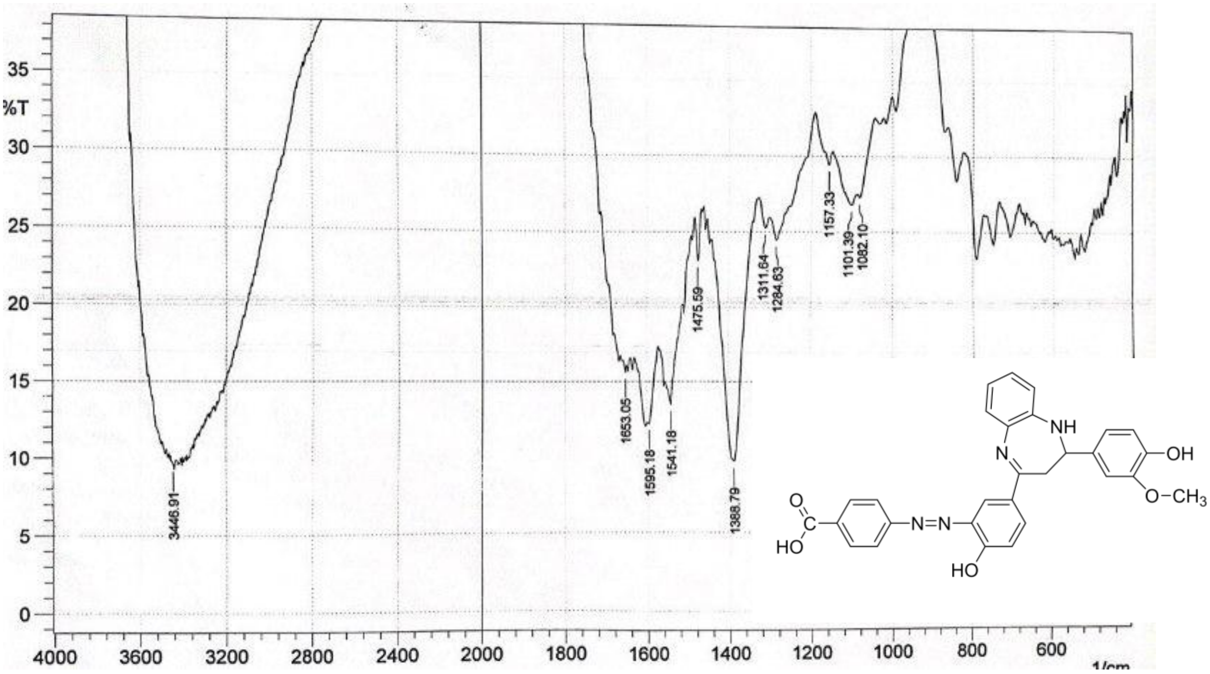


Figure (3-39) FT-IR Spectrum of compound[S14]

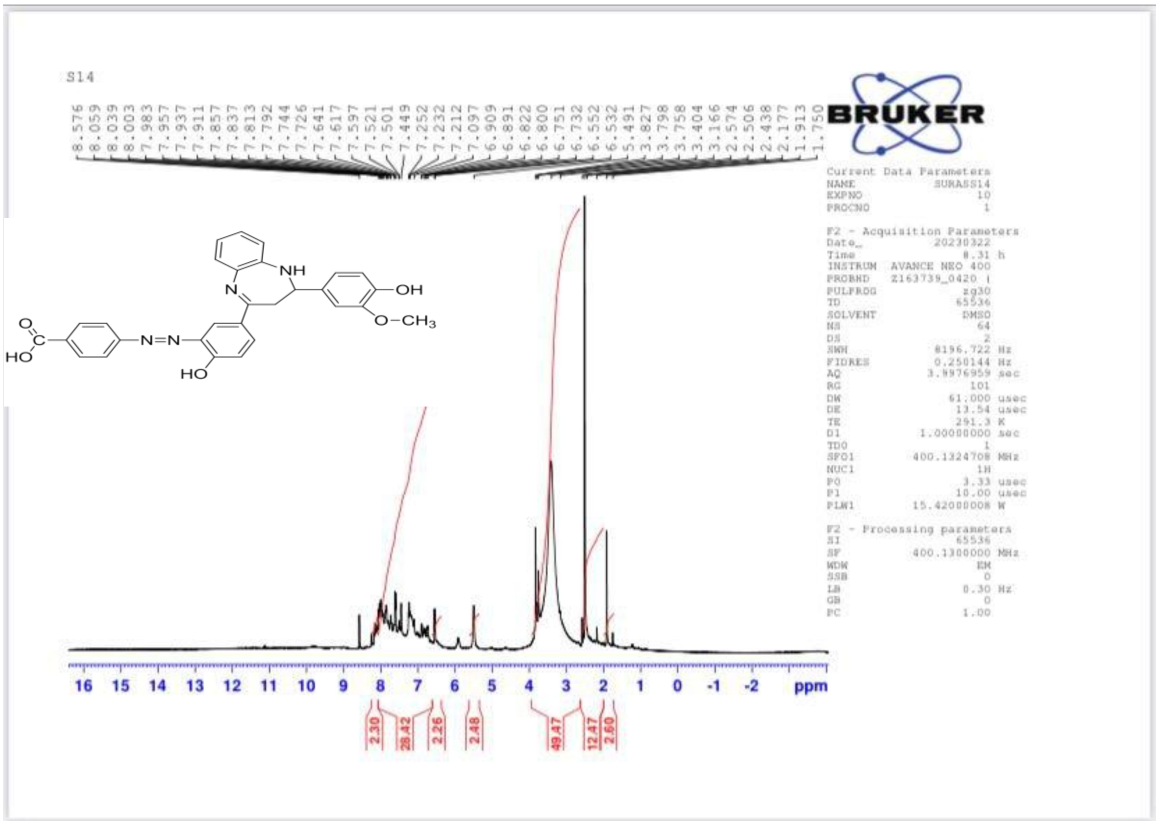
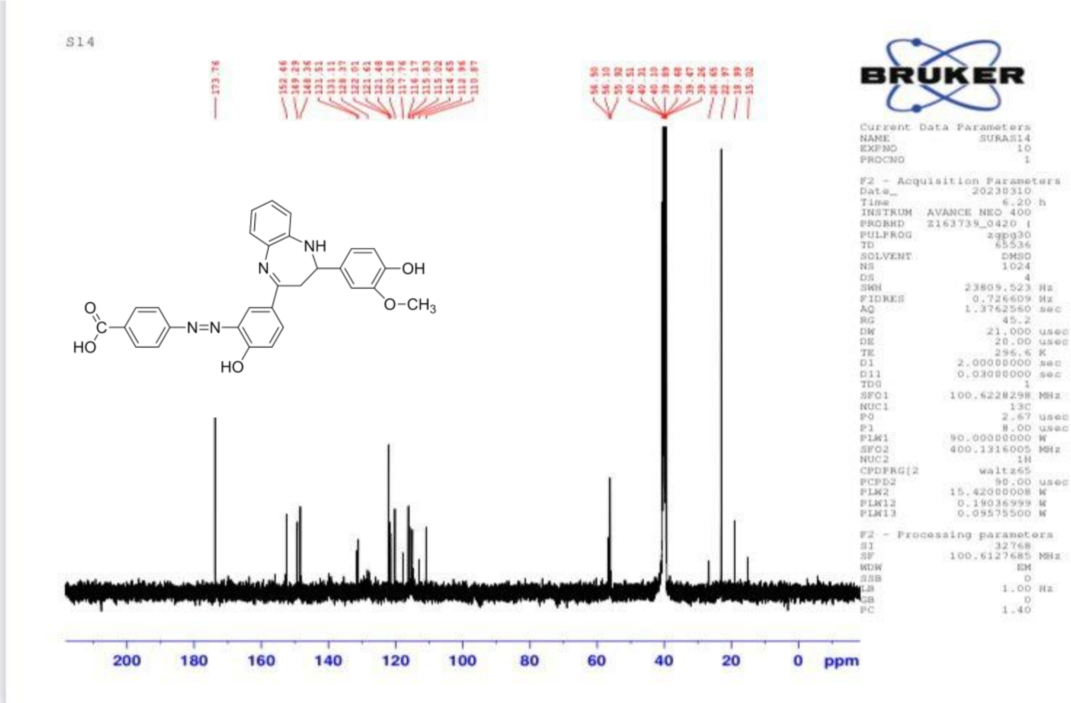
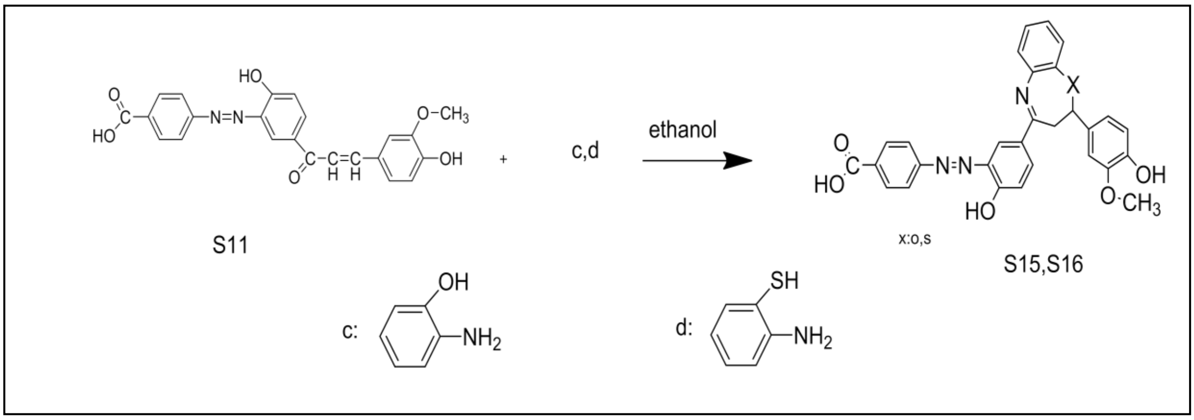


Figure (3-40) ^{1}H NMR of compound[S14]



Figure(3-41) ^{13}CNMR of compound[S14]



Equation 3-12: Synthesis of Compound [S15,S16]

The FTIR(v, cm^{-1}) spectrum of compound [S15] exhibited absorption band at (1593) cm-1 for N=N , (3481) cm^{-1} for O-H, C-H Ar (3101), C=O $_{\text{carboxylic acid}}$ (1691) , N-H (3267) .

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 5.8 for (H,OH) and signal at 1.9 for (H,CH $_{\text{Endo cyclic}}$) , (10H,Ar-H) : 6.12-7.6.

.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at 111-140 for : (18C,Ar-C), and signal at 56.12 for, C-H $_{\text{alpha}}$, (140) : C-OH, (85.01,39.3) :2C,C-H $_{\text{Endo cyclic}}$.

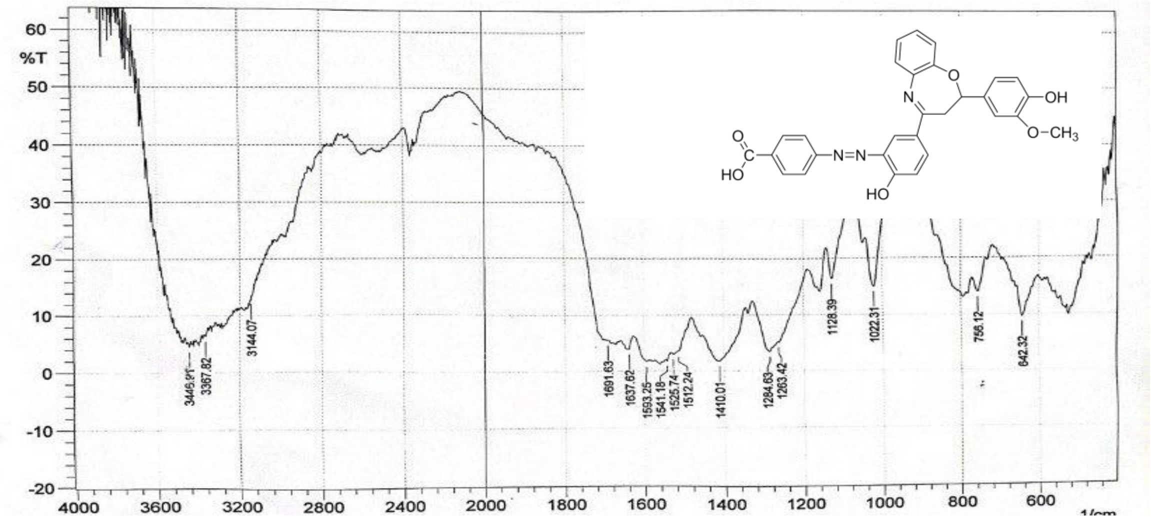


Figure (3-42) FT-IR Spectrum of compound[S15]

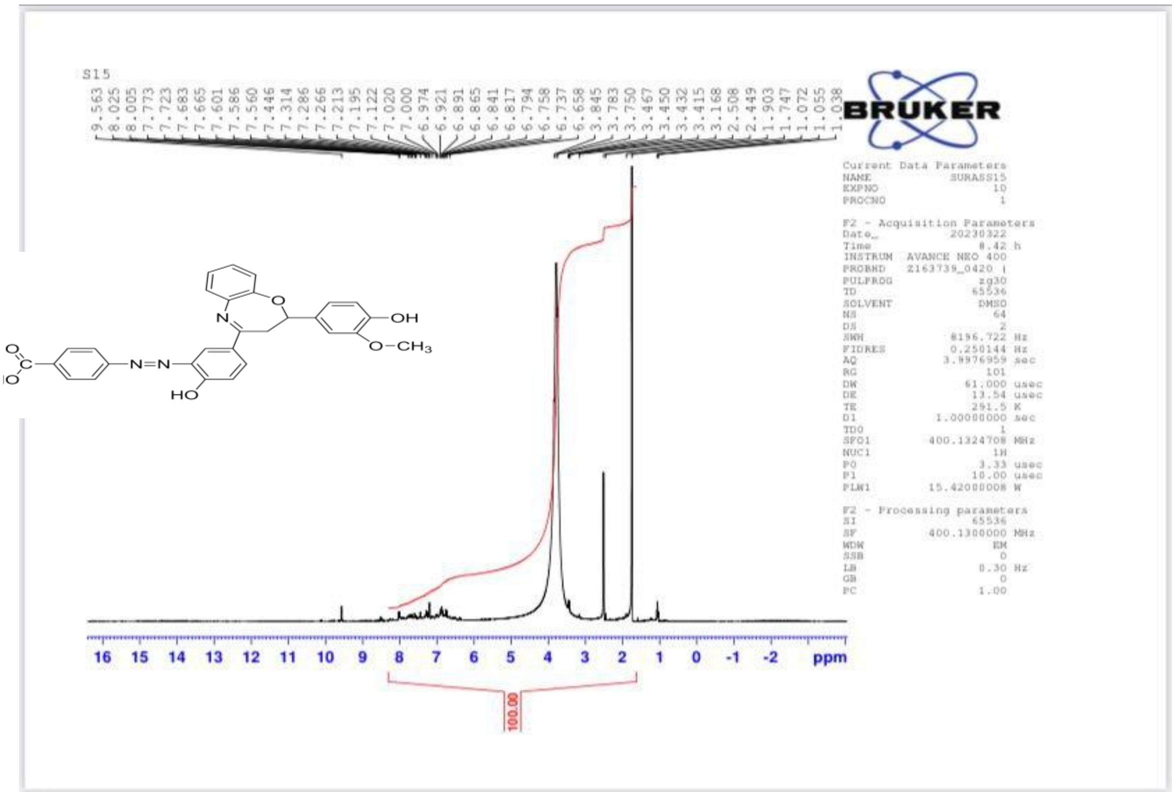
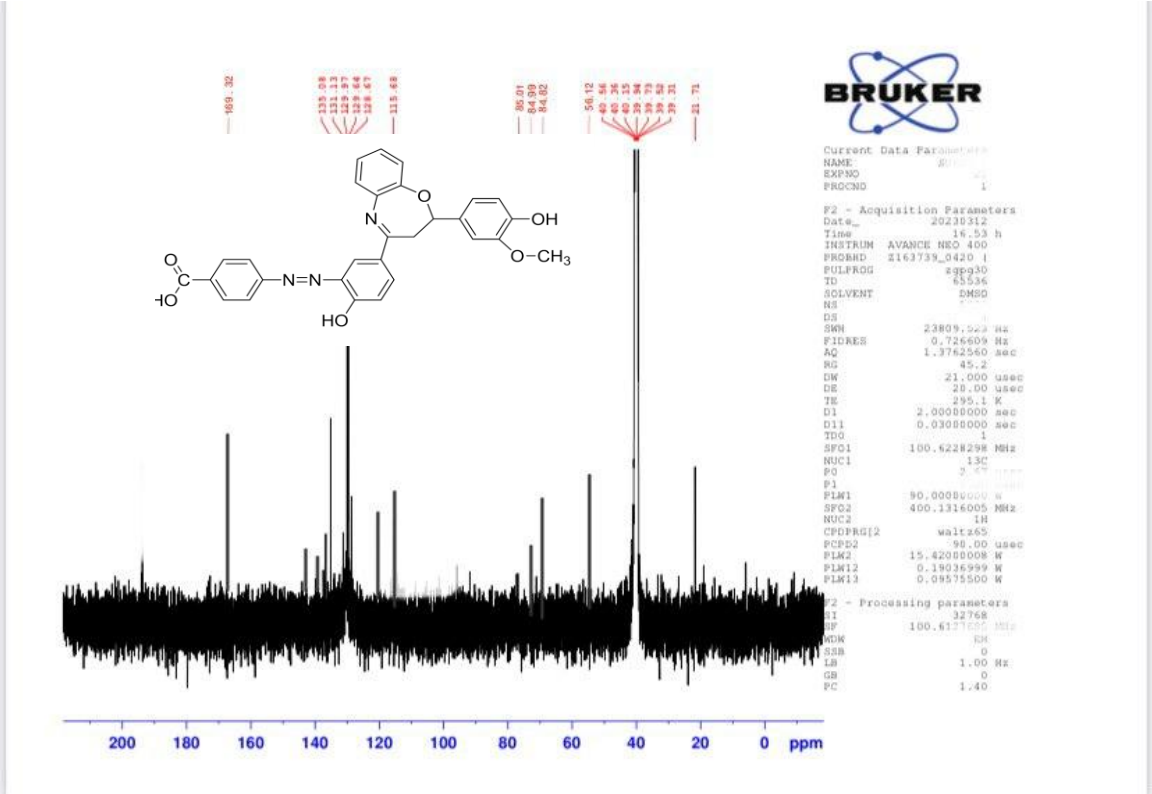


Figure (3-43) ^{1}H NMR of compound[S15]



Figure(3-44) ^{13}CNMR of compound[S15]

The FTIR (*v*, cm^{-1}) spectrum of compound [S16] exhibited absorption band at (1595) cm^{-1} for N=N.(1670) cm^{-1} for C=O $_{\text{Carboxylicacid}}$, O-H (3423), C-H Ar (3101),C=N (1653).

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 6.4 for (H,OH) and signal at 1.7 for (H,CH $_{\text{Endocyclic}}$), (10H,Ar-H) : 6.7-8.06
.
^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at 110-151.8 for (18C,Ar-C), and signal at 56.07 for ,C-H $_{\text{alpha}}$, (145.5) : C-OH , (168.2):C,COOH ,(39.1) :C,C-H $_{\text{Endocyclic}}$.

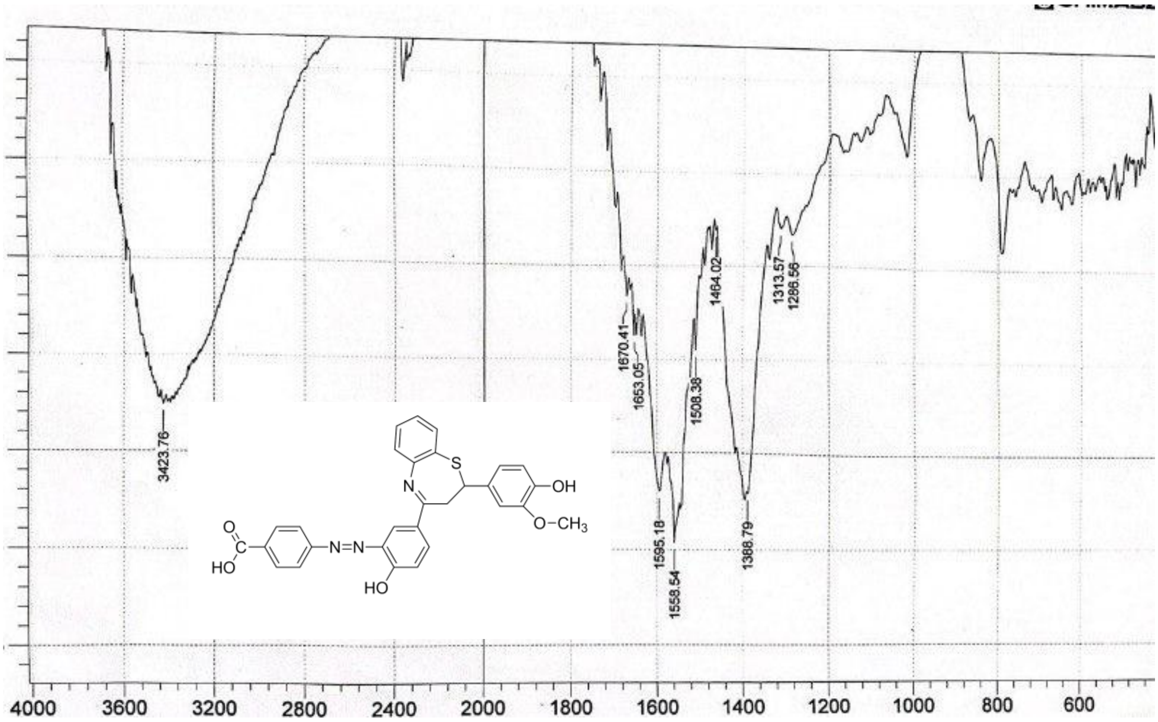


Figure (3-45) FT-IR Spectrum of compound[S16]

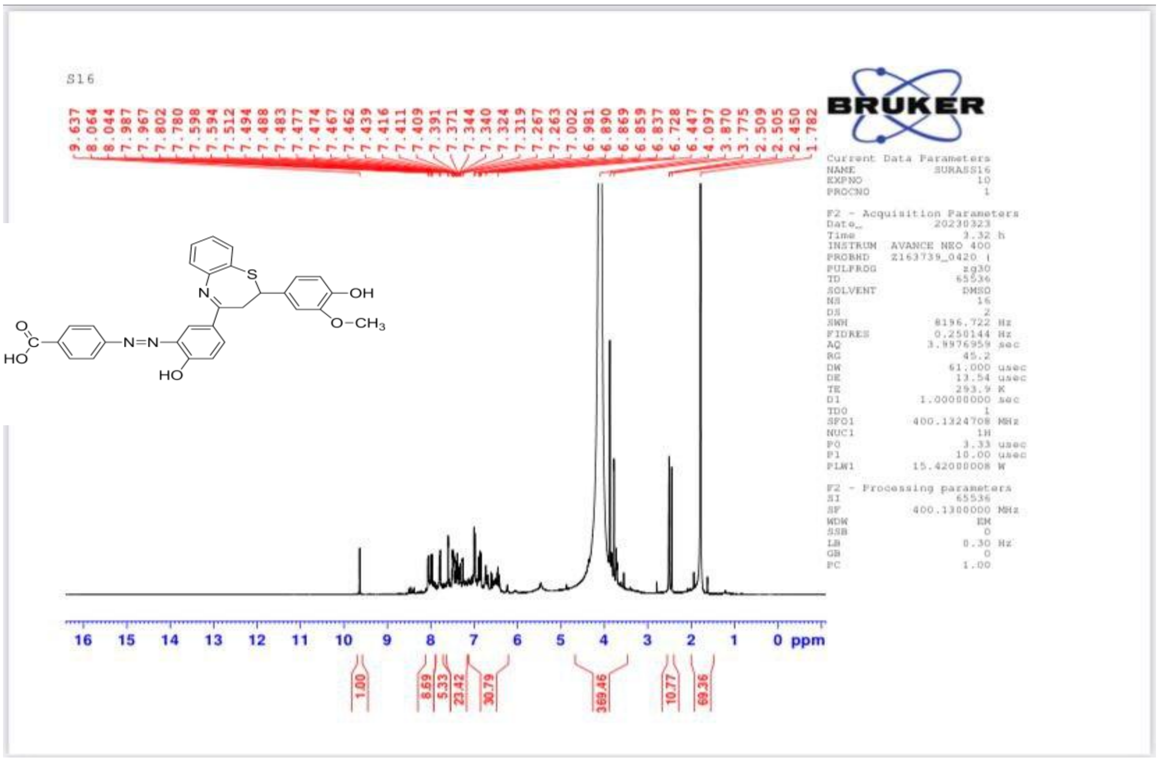
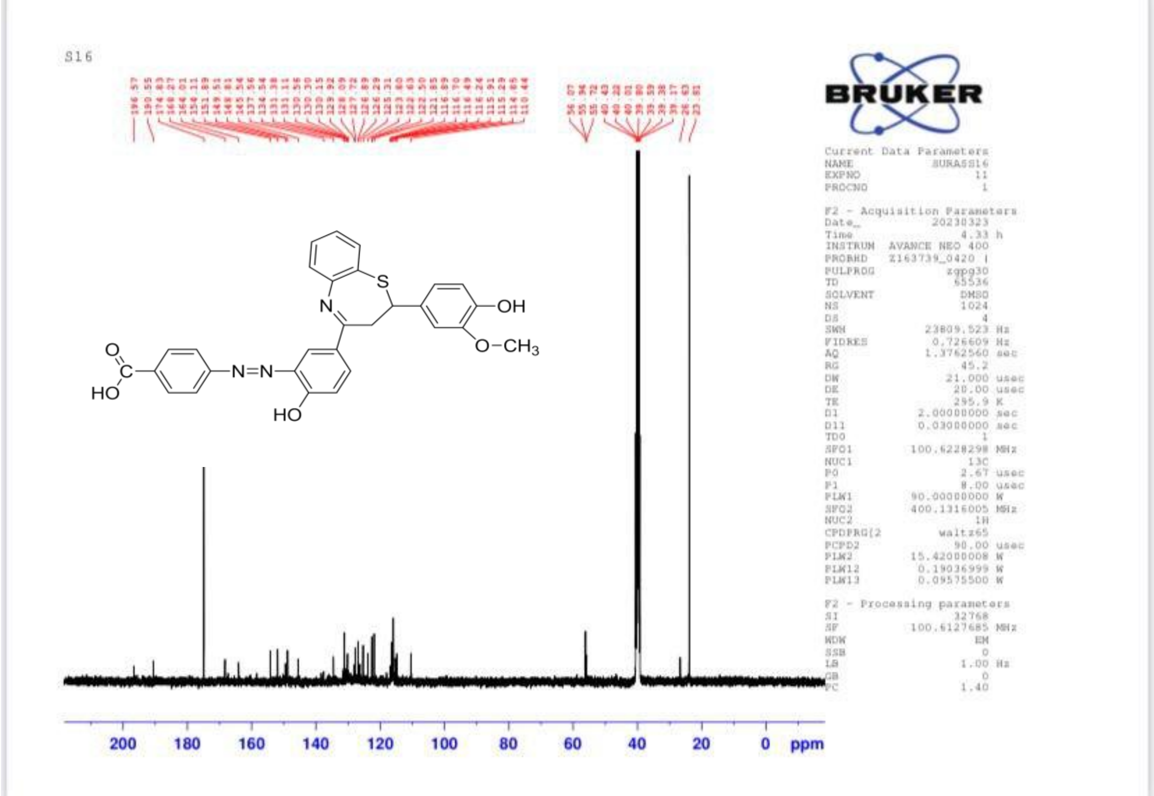


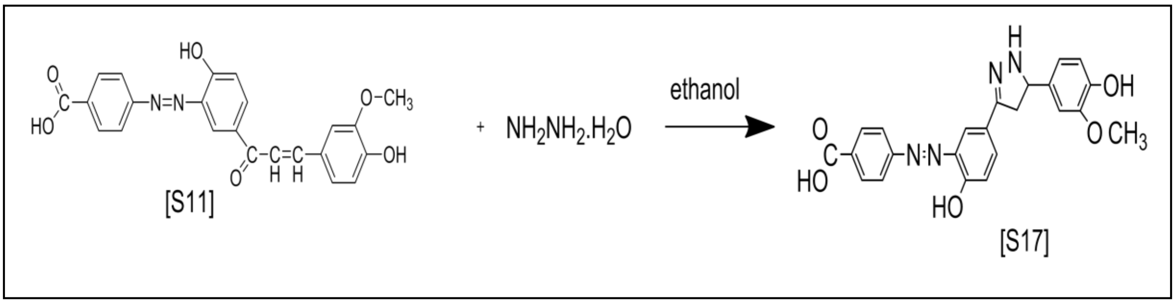
Figure (3-46) ^{1}H NMR of compound[S16]



Figure(3-47) ^{13}CNMR of compound[S16]

3.12 Synthesis of compounds [S17,S18]

(Pyrazol S17, Isoxazol S18) These compounds with a five-ring were prepared from the interaction of (hydrazine hydrate, hydroxylamine hydrochloride) compounds with ethanol in the presence of a few drops of sodium hydroxide with the presence of a reflex reaction. Paraffin oil was used during the reflex process so that the heat is distributed evenly on all parts of the round flask. These two reactions formed five heterogeneous cyclic compounds.



Equation 3-12: Synthesis of Compound [S17]

The FTIR spectrum of compound [S17] exhibited absorption band at (1550) cm^{-1} for N=N
IR (*v*, cm-1):(1600) cm^{-1} for $C=O_{\text{Crboxylicacid}}$,O-H (3383),C-H Ar (3200),

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 7.10 for N-H ,3.7 for $C\text{-}H_{\text{Alpha .}}$ (10H,Ar-H) : 6.6-8.05 ,($C\text{-}H_{\text{Endocyclic}}$) : 1.6.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at (107-151.8) for 18C,Ar-C, and signal at 56.0 for,$C\text{-}H_{\text{alpha}}$, (145.5) : C-OH , (168.2): C,COO ,(39.12) :C,$C\text{-}H_{\text{Endocyclic}}$.

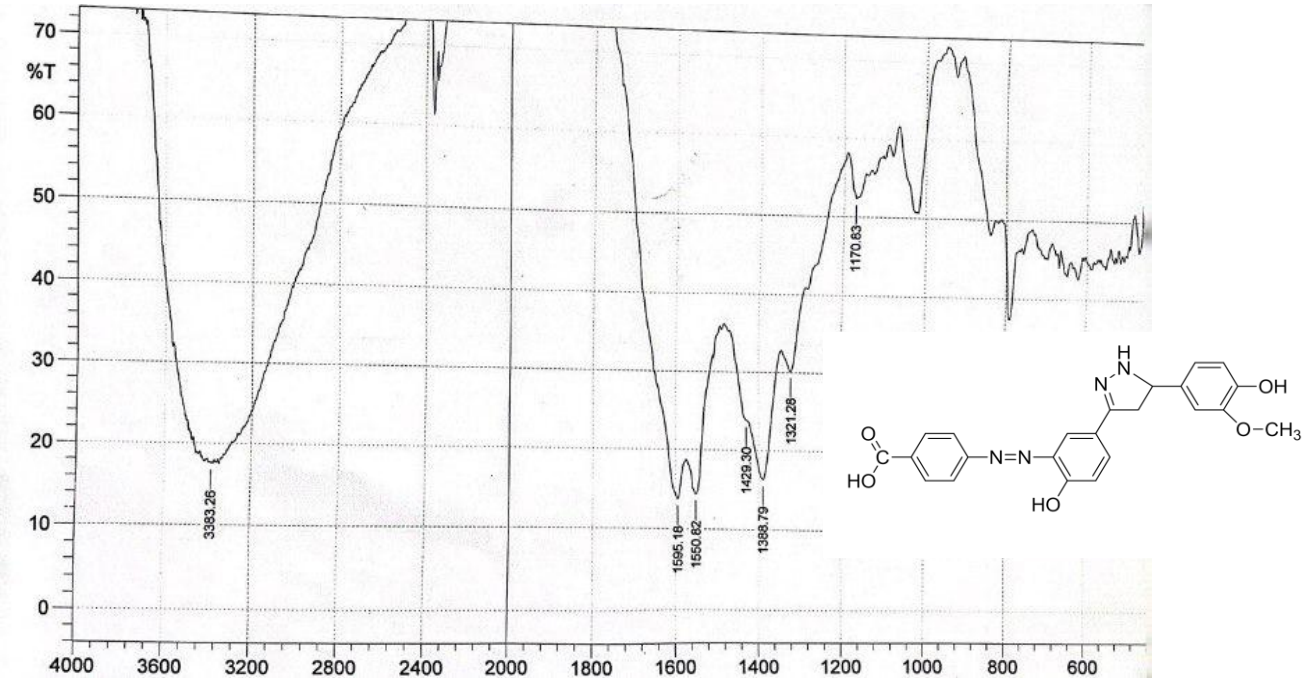


Figure (3-48) FT-IR Spectrum of compound[S17]

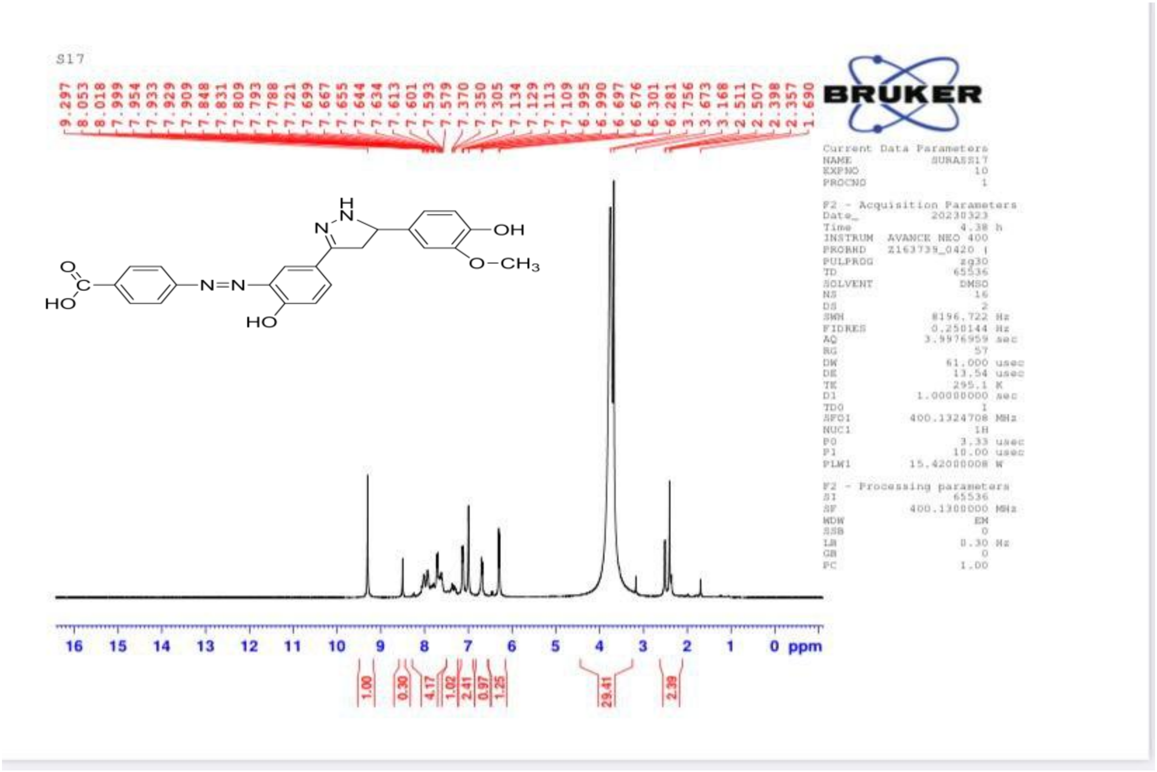
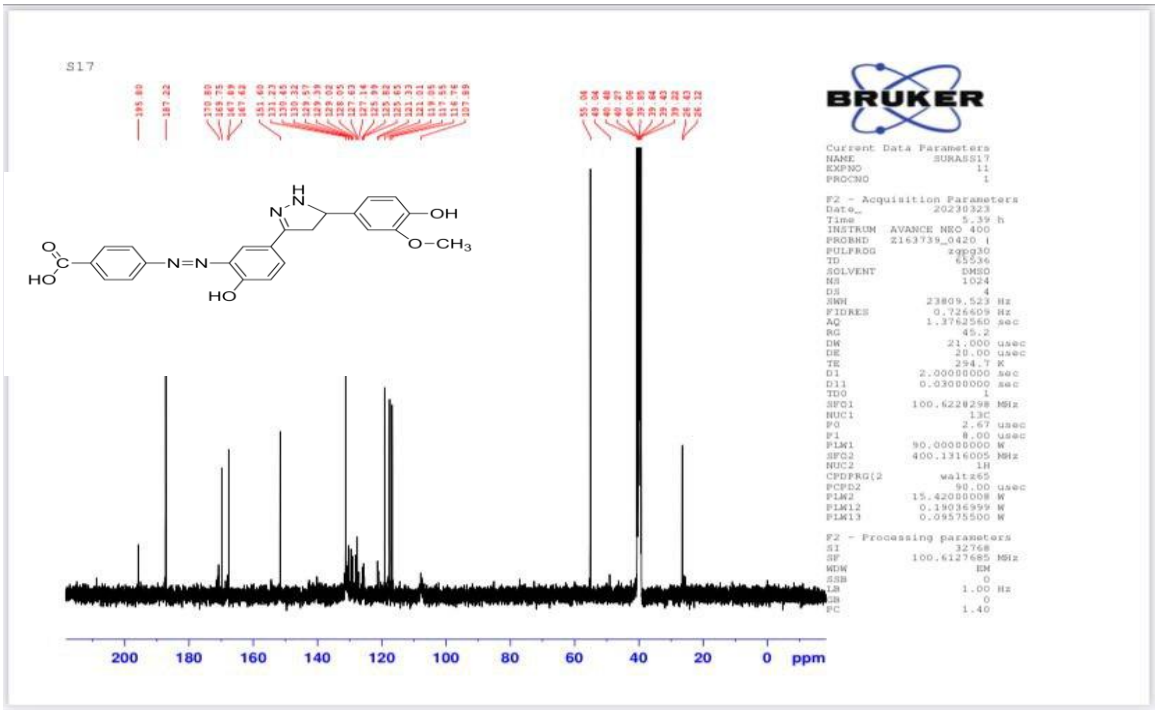
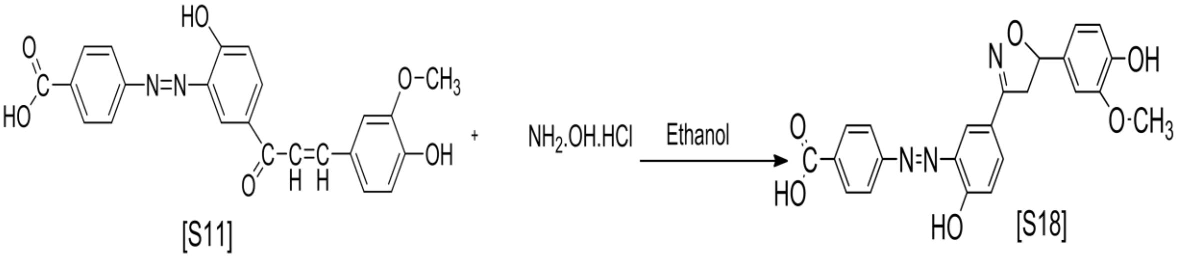


Figure (3-49) ^{1}H NMR of compound[S17]



Figure(3-50) ^{13}CNMR of compound[S17]



Equation 3-13: Synthesis of Compound [S18]

The FTIR (v, cm^{-1}): spectrum of compound [S18] exhibited absorption band at (1541) cm^{-1} for N=N ,(1654) cm^{-1} for C=O $_{\text{Crboxylicacid,}}$O-H (3426),C-H Ar (3200).

13C-NMR(100 MHz,DMSO-d6) (δ, ppm) : (110-132) : (18C,Ar-C), (145.5) : C-OH , (169): C,COO, (55.88): C-H $_{\text{Alpha}}$,(85.12,39.34) :2C,C-H $_{\text{Endocyclic}}$.

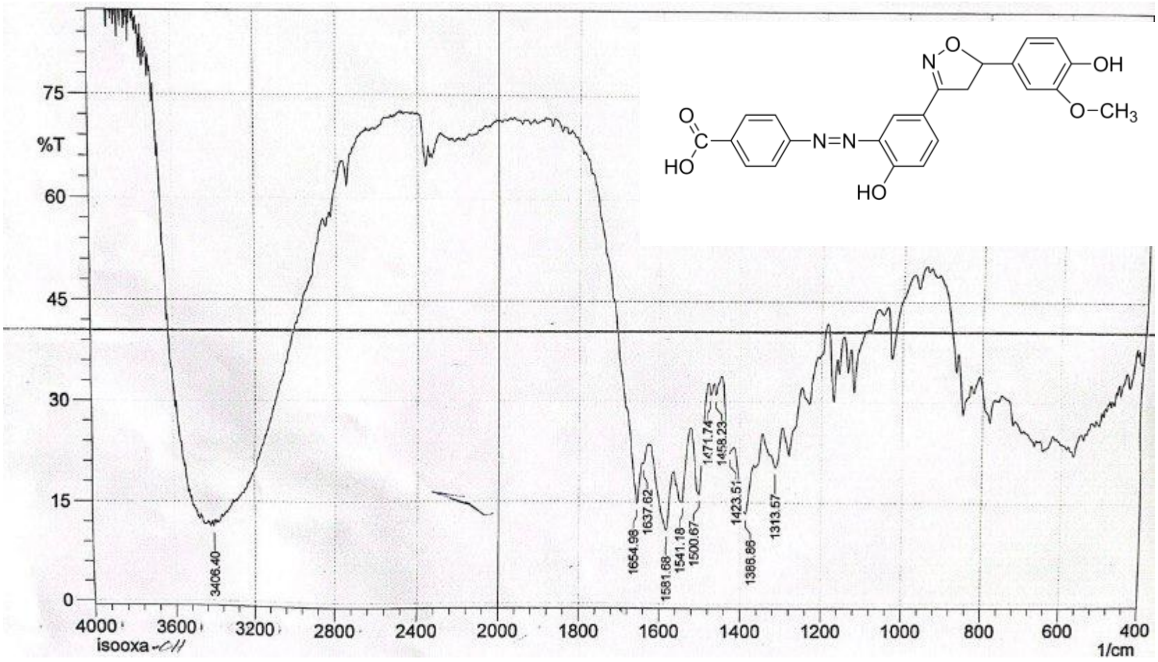
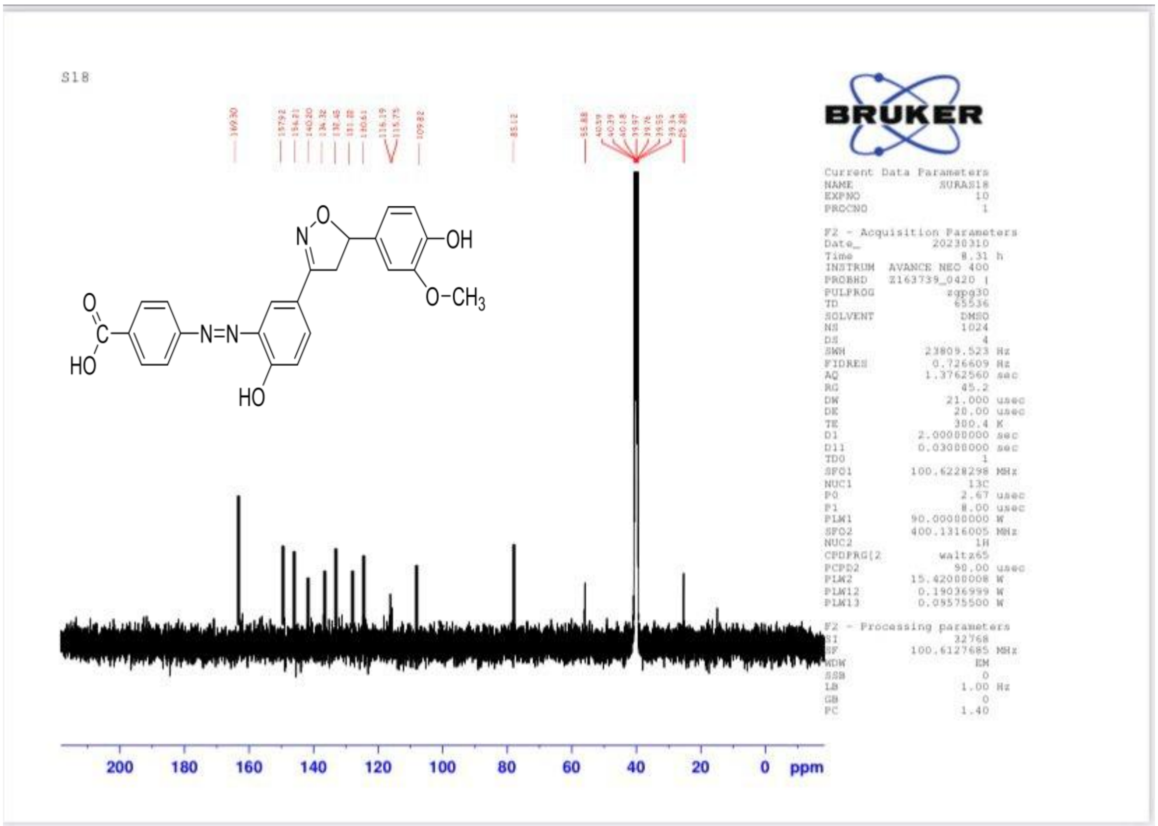


Figure (3-51) FT-IR Spectrum of compound[S18]



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DS                       4
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FIDRES            0.726609 Hz
AQ               1.3762560 sec
RG                    45.2
DW                  21.000 usec
DE                   20.00 usec
TE                   300.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1
SFO1           100.6228298 MHz
NUC1                   13C
P0                    2.67 usec
P1                    8.00 usec
PLW1           90.00000000 W
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz65
PCPD2                90.00 usec
PLW2           15.42000008 W
PLW12           0.19036999 W
PLW13           0.09575500 W

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SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

Figure (3-52) ^{13}CNMR of compound[S18]

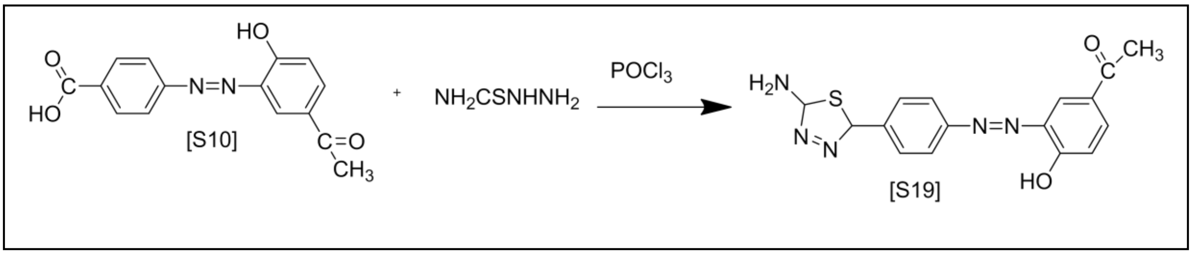
3.13 Synthesis of compound [S19]

The azo dye was reacted with a thiosemicarbazide compound in the presence of $POCl_3$ to form a thiadiazole compound, and the evidence for this is the appearance of band for NH_2 at (3466-3327) according to the FT-IR technique, ^{1}HNMR show the signal at(6.8) for H-Aroma. C-H_{Alpha} ,(39.3) for C-NH_2

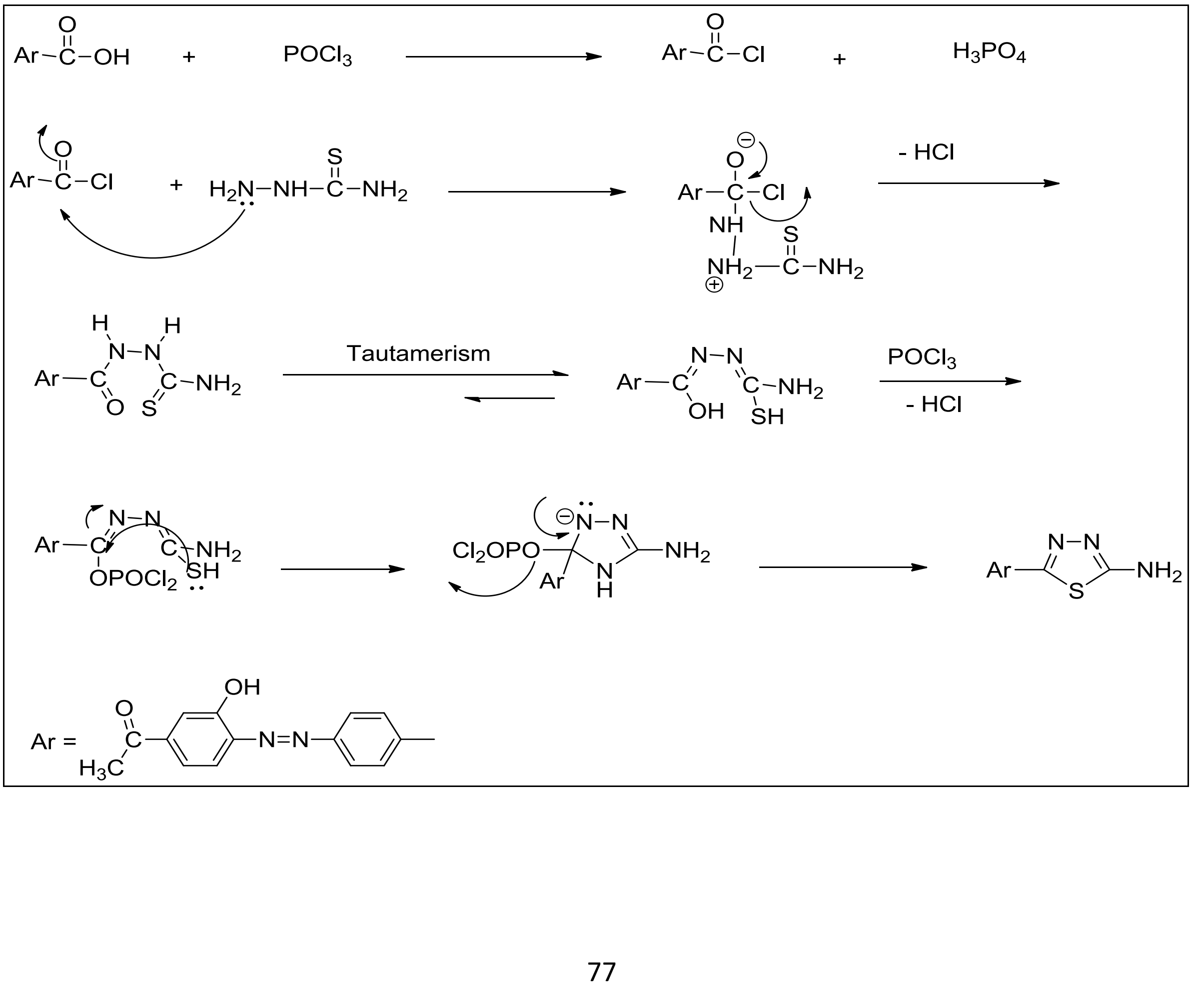
FT-IR (v, cm^{-1}): C-H $_{Ar}$ (3090), C=O $_{keton}$ (1600), O-H (3437), N=N (1583).

^{1}H-NMR (400 MHz,DMSO-d6) :(δ, ppm) (C-H_{Alpha}): 2.5, (7H,Ar-H) : 6.8-7.8.

^{13}C-NMR (100 MHz,DMSO-d6):(δ, ppm) (C-H_{Alpha}): 26, (12C,Ar-C) : (115-131), (C=O): 196 .



Equation 3.14 synthesis of compound[S19]



Scheme (1) Mechanism synthesis of the compound [S19][122]

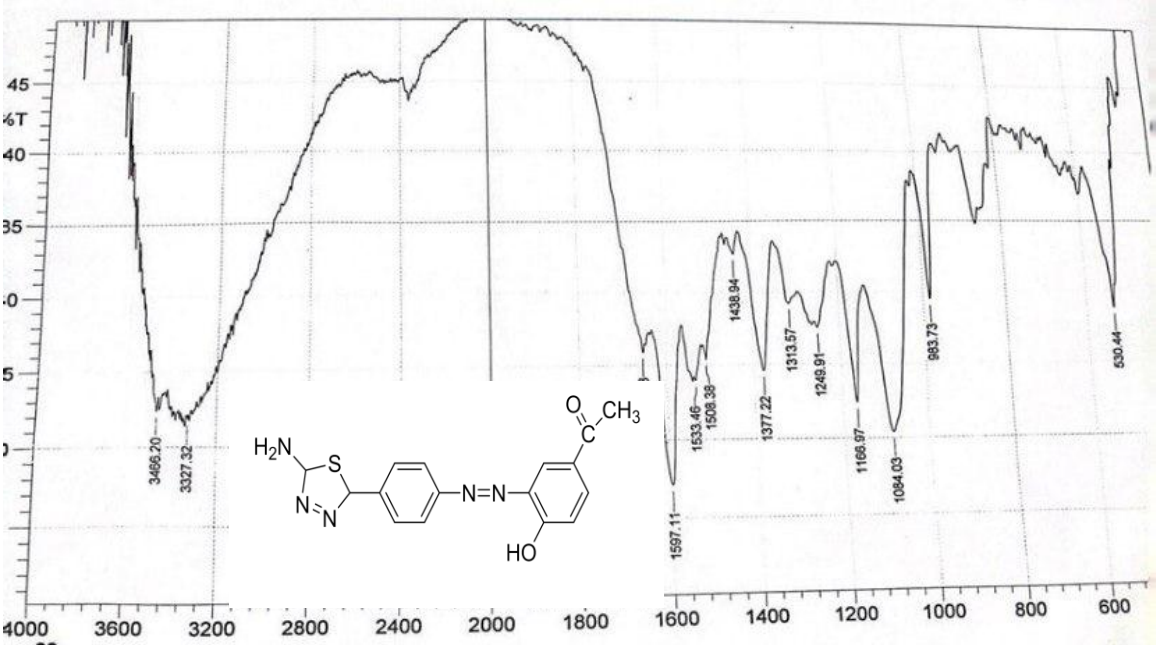
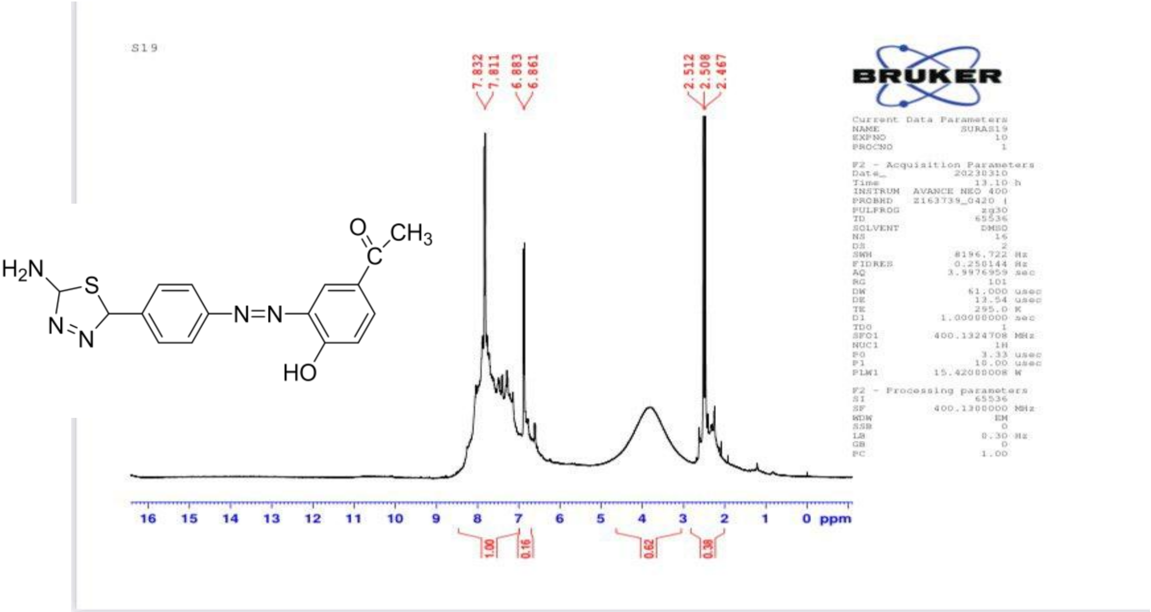
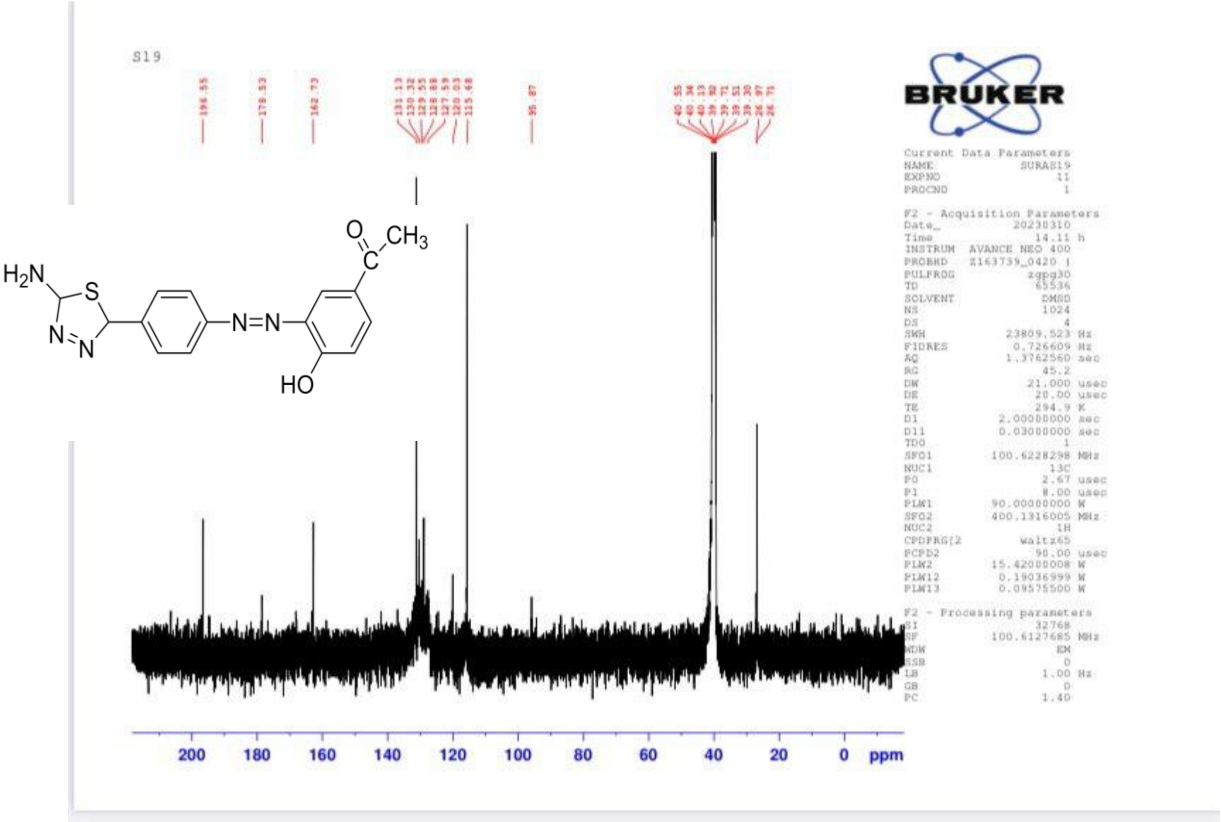


Figure (3-53) FT-IR Spectrum of compound[S19]



Figure(3-54) ^{1}HNMR of compound[S19]



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DE                20.00 usec
TE                294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
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P0                 2.67 usec
P1                 8.00 usec
PLW1        90.00000000 W
SFO2        400.1316005 MHz
NUC2                 1H
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F2 - Processing parameters
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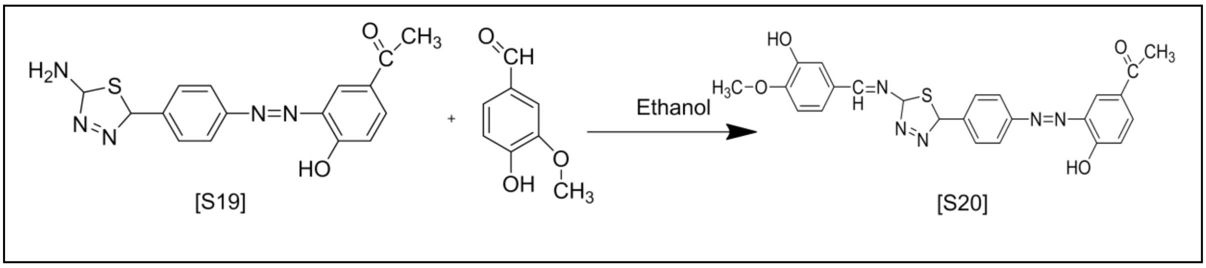
Figure (3-55)^{13}CNMR of compound [S19]

3.14 Synthesis of compound [S20]

Thiadiadiazole reacts with aldehyde(vanillin) to form a Schiff base. According to the FT-IR technique, the amine group disappeared(3466-3327) and the C=N group appeared at(1664), (1600):C=O $_{\text{Ketone}}$.



Equation 3.15 synthesis of compound[S20]

The FTIR(*v*, cm^{-1}): spectrum of compound [S20] exhibited absorption band at (1664) cm^{-1} for C=N,O-H (3200),C-H Ar (3090), , N=N (1583).

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 2.5 for $C-H_{Alpha}$, 5.35 for O-H, (2H,$C-H_{Endocyclic}$) : 3.8,4.01 , ($CH=N_{Schiff}$) : 7.93, (H,CH_{Arom}) :6.6-7.7.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at 197.4 for $C=O_{Keton}$ and signal at 21.89 for,$C-H_{alpha}$, (111-140) : (18C,Ar-C), (148.7) : C-OH , (197.4):$C=O_{Keton}$,

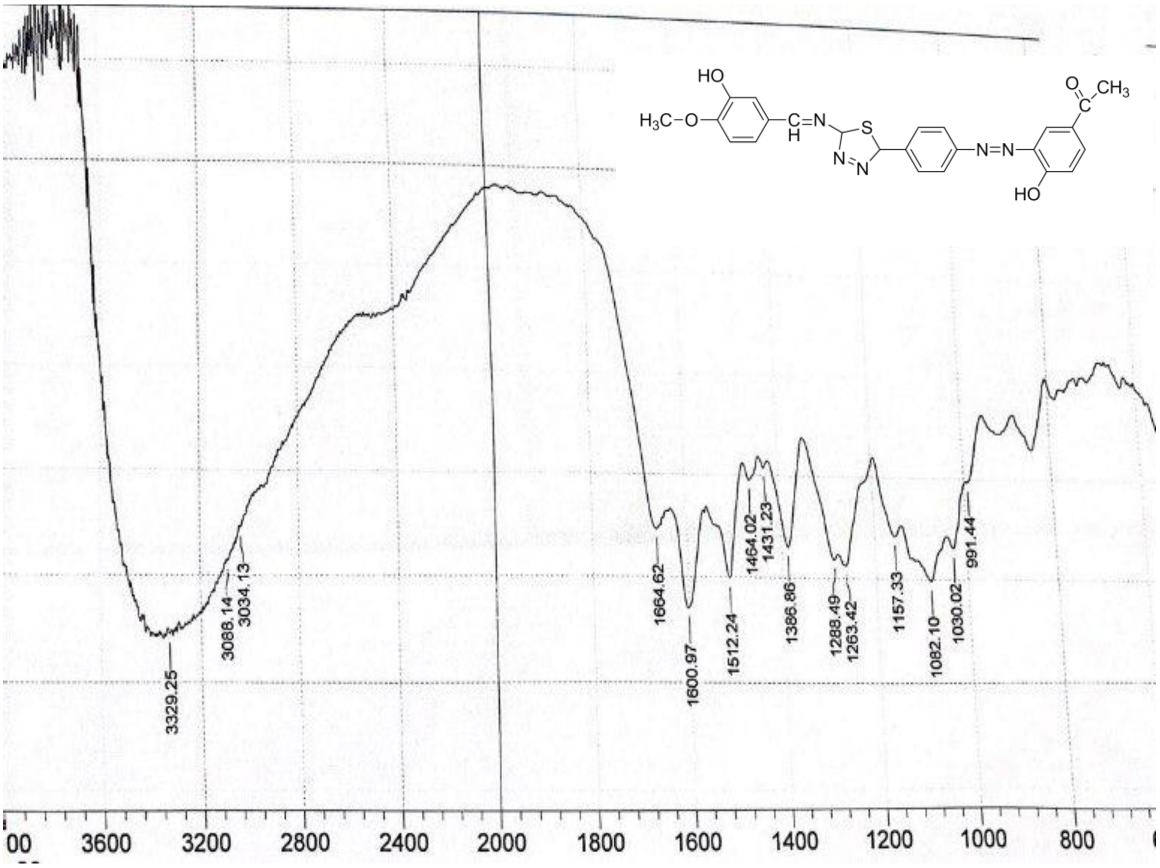
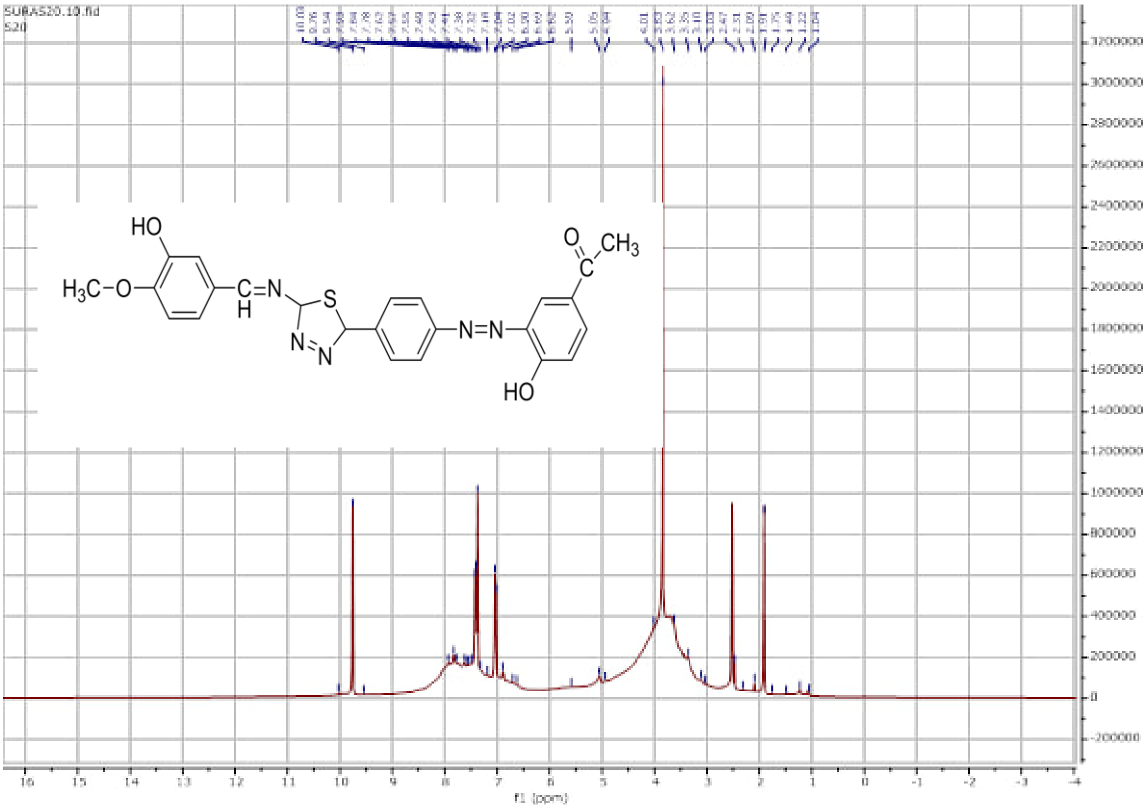
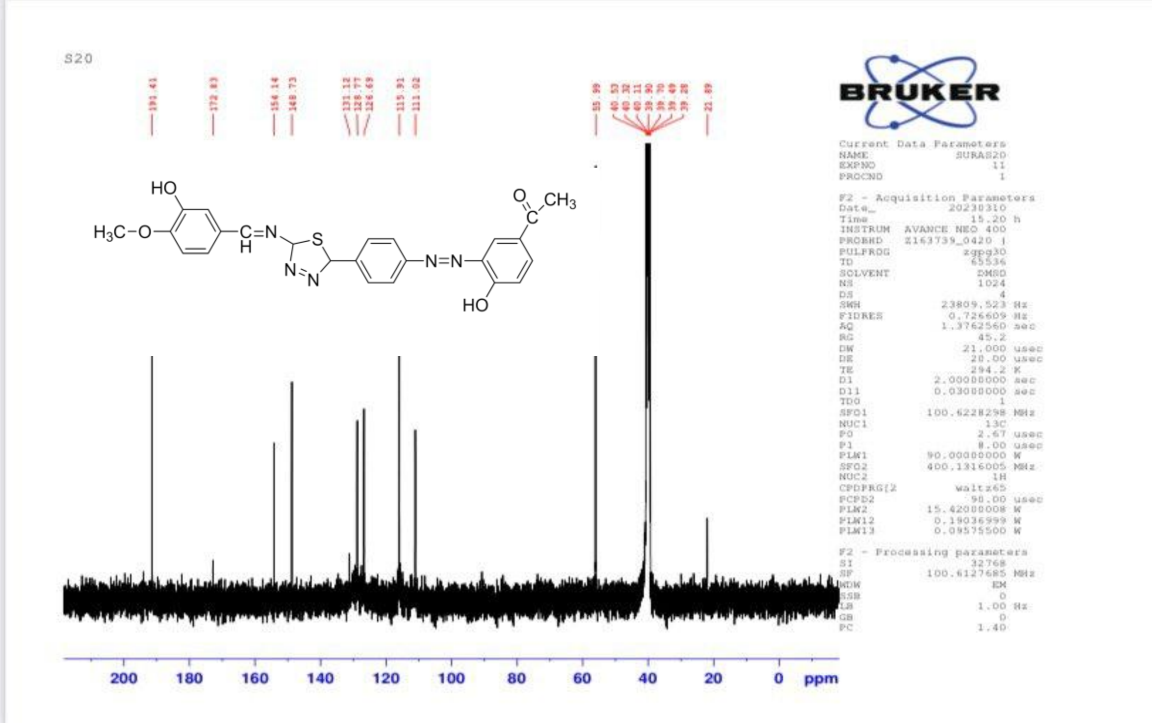


Figure (3-56) FT-IR Spectrum of compound[S20]



Figure(3-57) ^{1}HNMR of compound[S20]



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PROCNO                1

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TD                65536
SOLVENT            DMSO
NS                 1024
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ            1.3762560 sec
RG                 45.2
DW               21.000 usec
DE                20.00 usec
TE                294.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
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P0                 2.67 usec
P1                 8.00 usec
PLW1        90.00000000 W
SFO2        400.1316005 MHz
NUC2                 1H
CPDPRG[2        waltz65
PCPD2             90.00 usec
PLW2        15.42000008 W
PLW12        0.19036999 W
PLW13        0.09575500 W

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WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Figure (3-58)^{13}CNMR of compound [S20]

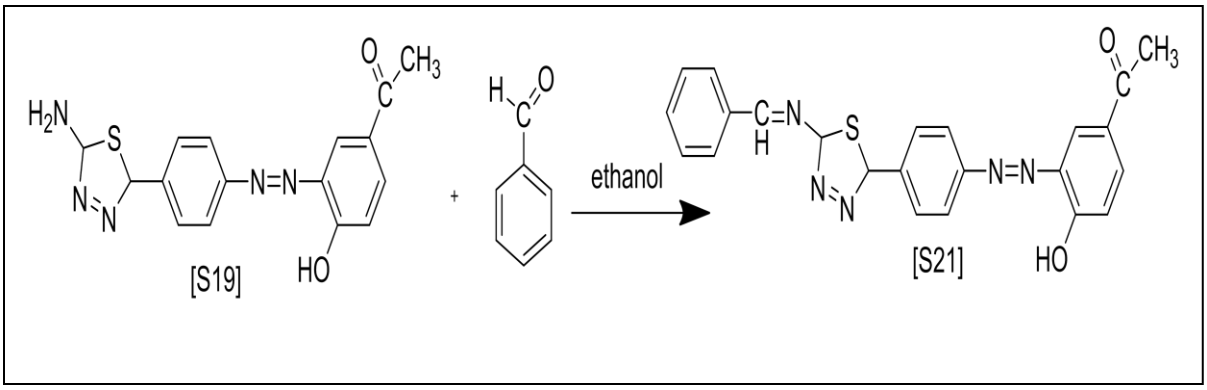
3.15 Synthesis of compound [S21]

Thiadiadiazole reacts with aldehyde(benzaldehyd) to form a Schiff base. According to the FT-IR technique, the amine group disappeared(3466-3327) and the C=N group appeared at(1654) ,(1604) for C=O$_{\text{keton}}$,(3100) for C-H$_{\text{Ar}}$.

The FTIR(*v*, cm^{-1}): spectrum of compound [S20] exhibited absorption band at (1604) cm^{-1} for C=N$_{\text{Schiff}}$,(1604) cm^{-1} for C=O$_{\text{ketone ,}}$ N=N (1541),

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 5.5 for O-H ,2.5 for C-H$_{\text{Alpha ,}}$ (12H,Ar-H) : 7.3-7.9 , (C-H$_{\text{Endocyclic}}$) : 3.58, (CH-N$_{\text{Schiff}}$) :8.6.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at 111-131.1 for (18C,Ar-C) and signal at 21.7 for, C-H$_{\text{alpha}}$, (135.5) : CH=N$_{\text{Schiff}}$



Equation 3.16 synthesis of compound[S21]

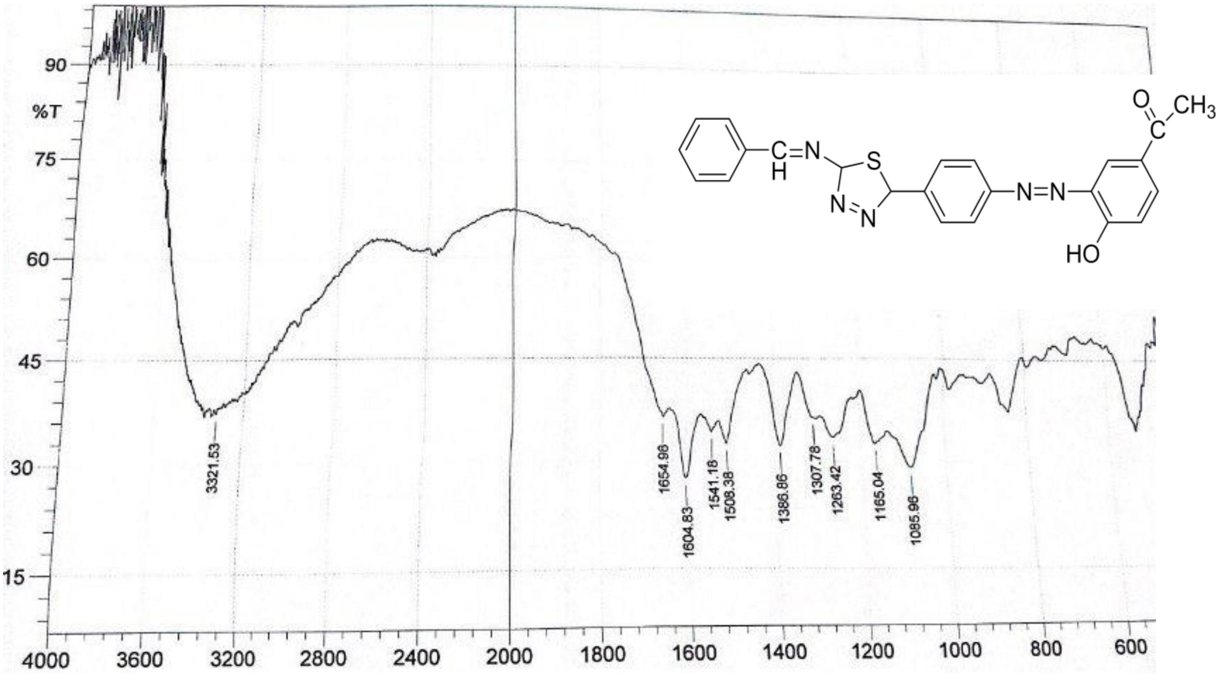
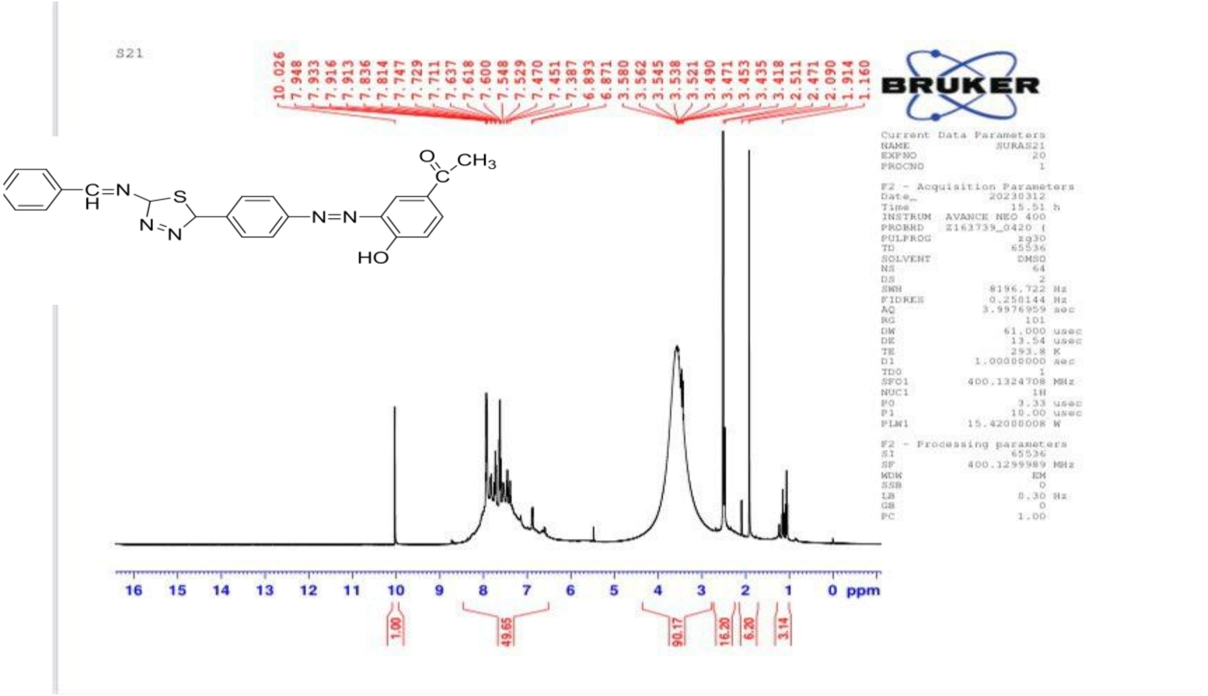


Figure (3-59) FT-IR Spectrum of compound[S21]



Figure(3-60) ^{1}HNMR of compound[S21]

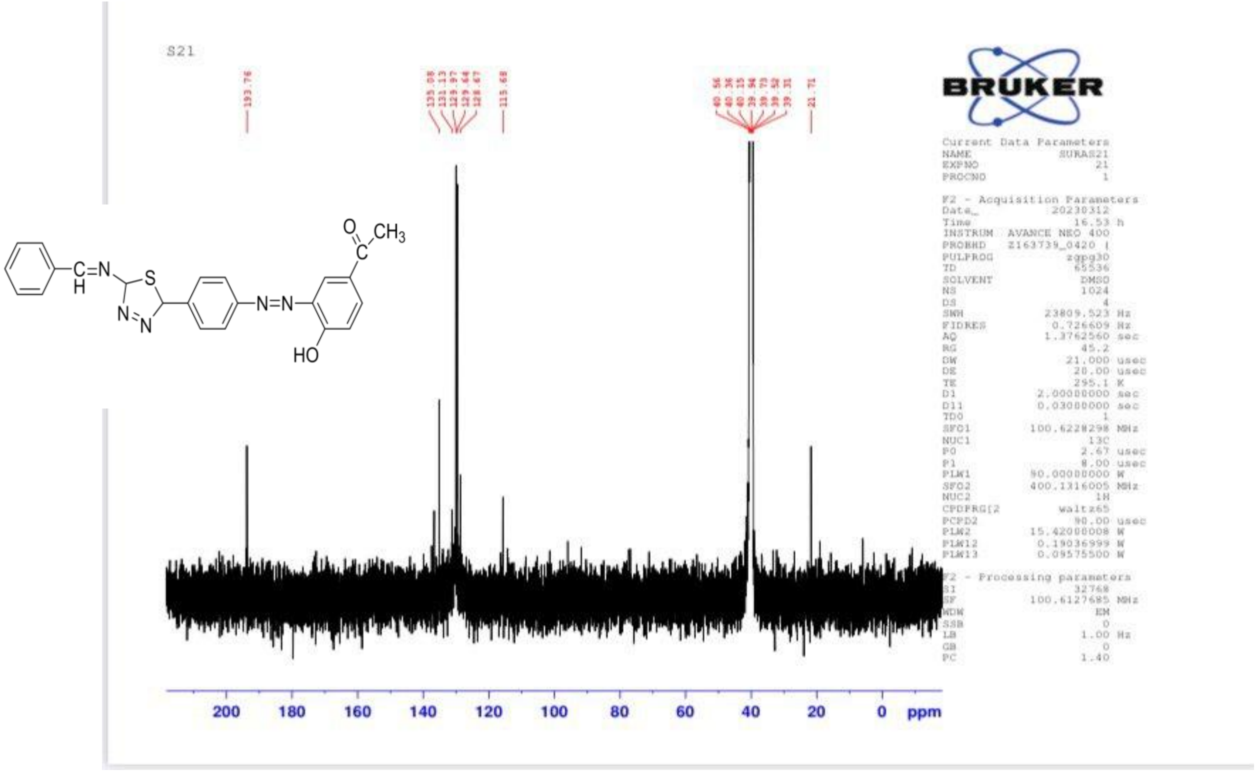


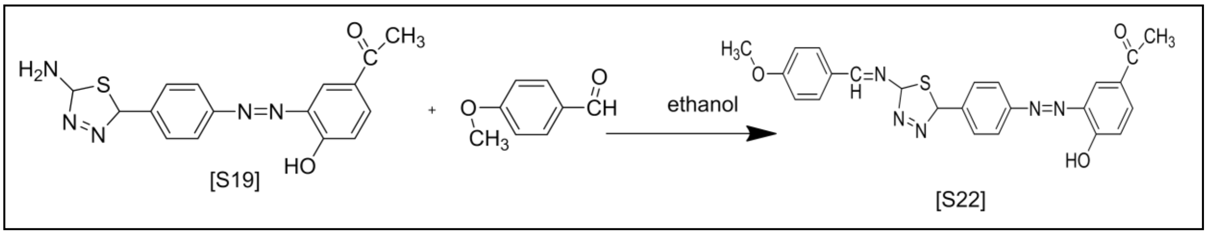
Figure (3-61)^{13}CNMR of compound [S21]

3.16 Synthesis of compound [S22]

Thiadiadiazole reacts with aldehyde(*P*-methoxy benzaldehyd) to form a Schiff base. According to the FT-IR technique, the amine group disappeared(3466-3327) and the C=N group appeared at(1647),(1604) for C=O$_{keton,}$ (1541) for N=N

The FTIR (*v*, cm^{-1}): spectrum of compound [S20] exhibited absorption band at (1647) cm^{-1} for C=N , O-H (3240),C-H $_{alpha}$ (2900) , (1604) cm^{-1} for C=O $_{Keton}$, N=N (1541) .

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 6.5-7.8 for (11H,Ar-H), for C-H $_{Alpha}$: 2.5, H,C-H$_{Schiff}$: 8.59 , (H,C-H $_{endocyclic}$) : 3.8,4.2



Equation 3.17 synthesis of compound[S22]

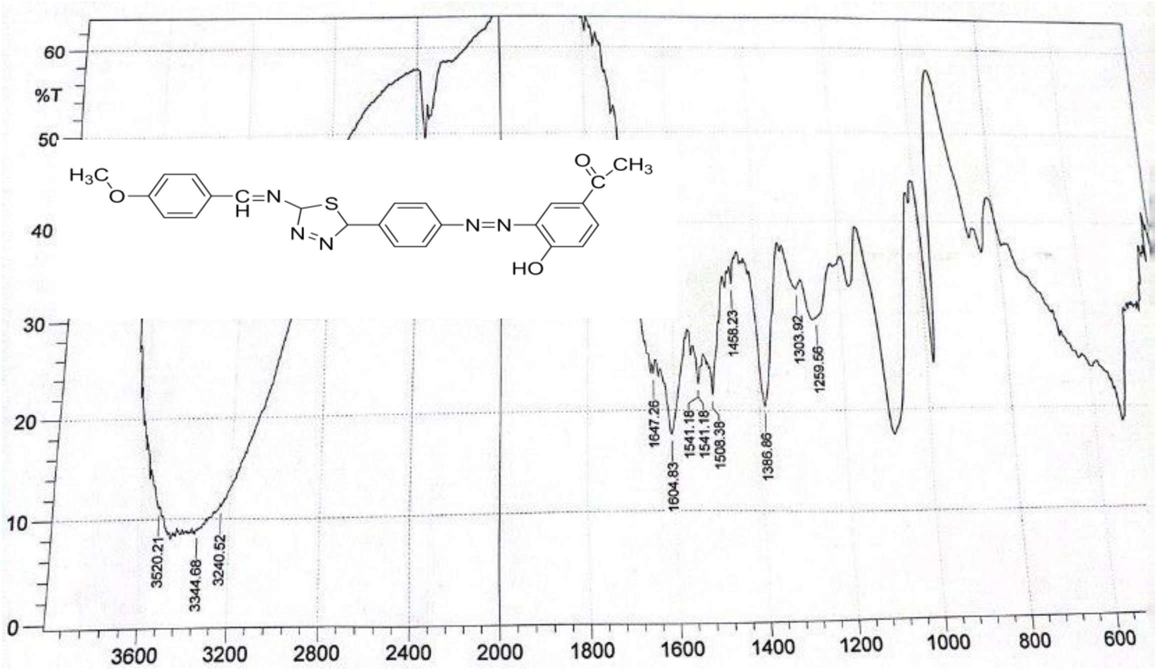
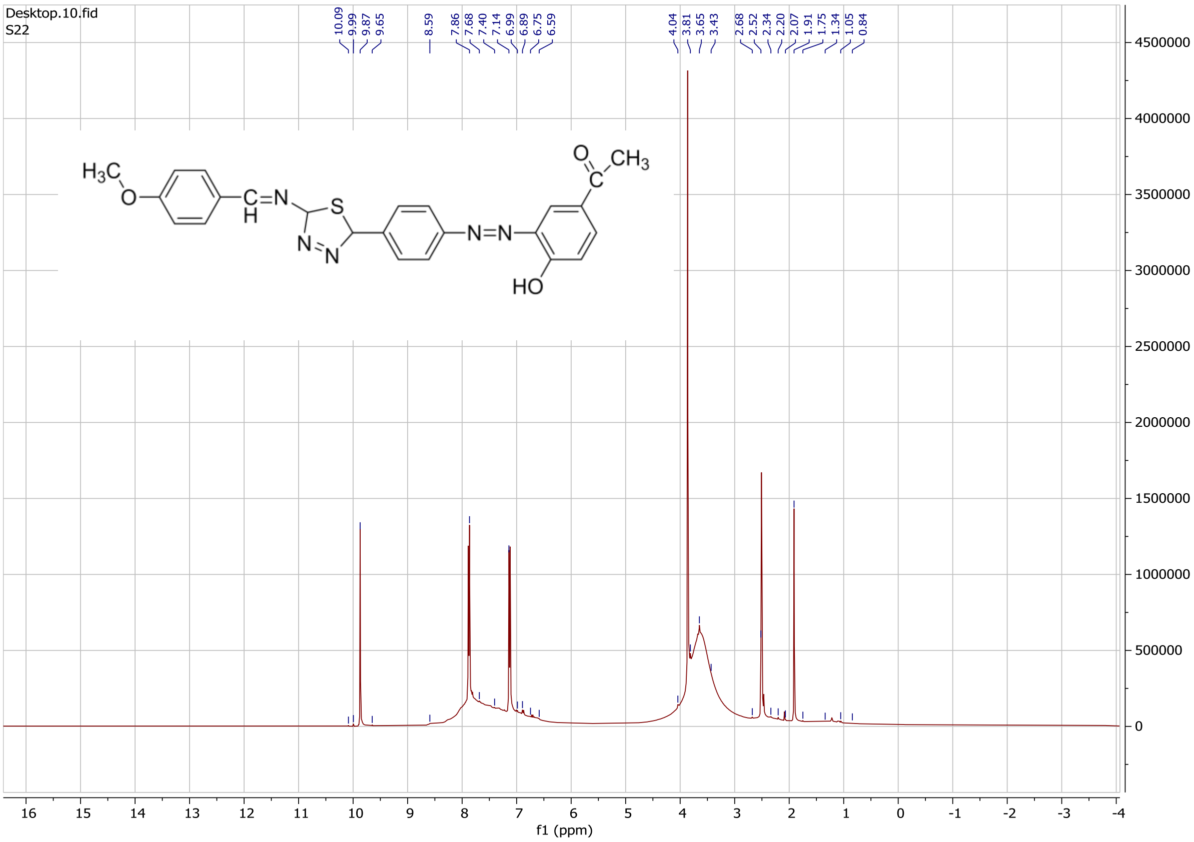


Figure (3-62) FT-IR Spectrum of compound[S22]



Figure(3-63) ^{1}HNMR of compound[S22]

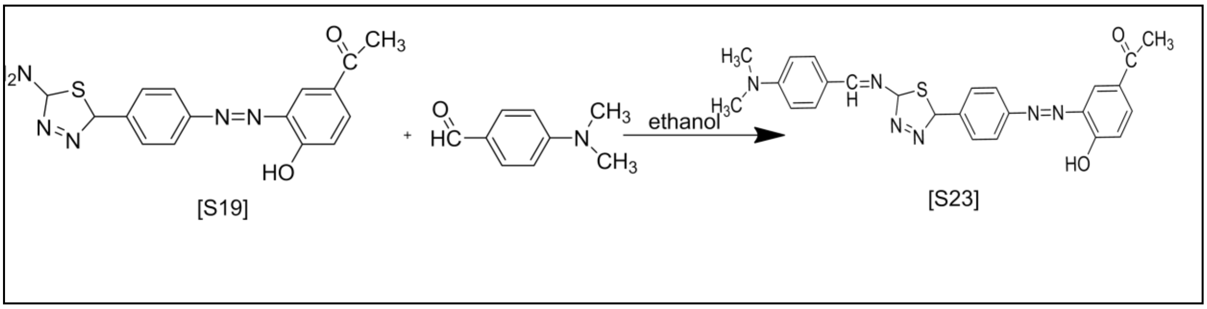
3.17 Synthesis of compound [S23]

Thiadiadiazole reacts with aldehyde(dimethyl aminobenzaldehyd) to form a Schiff base. According to the FT-IR technique, the amine group disappeared(3466-3327) and the C=N group appeared at(1670),(1600) for C=O_{keton} , (2810) for C-H_{Alpha}

The FTIR (*v*, cm^{-1}): spectrum of compound [S23] exhibited absorption band at (1670) cm-1 for CH=N_{Schiff} ,(1600) cm^{-1} for C=O_{ketonc} , C-H Ar (3100), C=O $_{ketonc}$ (1600), N=N (1541).

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 4.2 for C-H $_{Endocyclic}$,2.5 for C-H $_{Alpha}$, (11H,Ar-H) : 6.7-8.4 , H,C-H_{Schiff} :8.67 .

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at 111-131.8 for (18C,Ar-C), and signal at 26.6 for,C-H_{alpha} , (154.6) : C-OH , (163.1):CH=N $_{Schiff}$, C=O_{Ketone} (190).



Equation 3.18 synthesis of compound[S23]

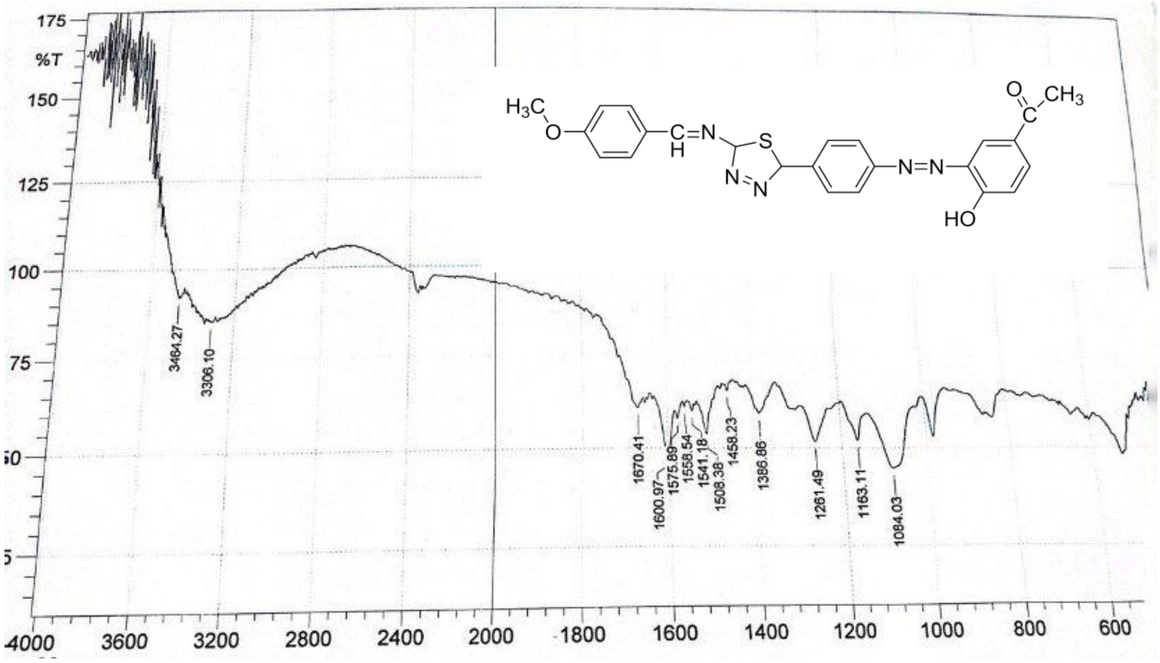
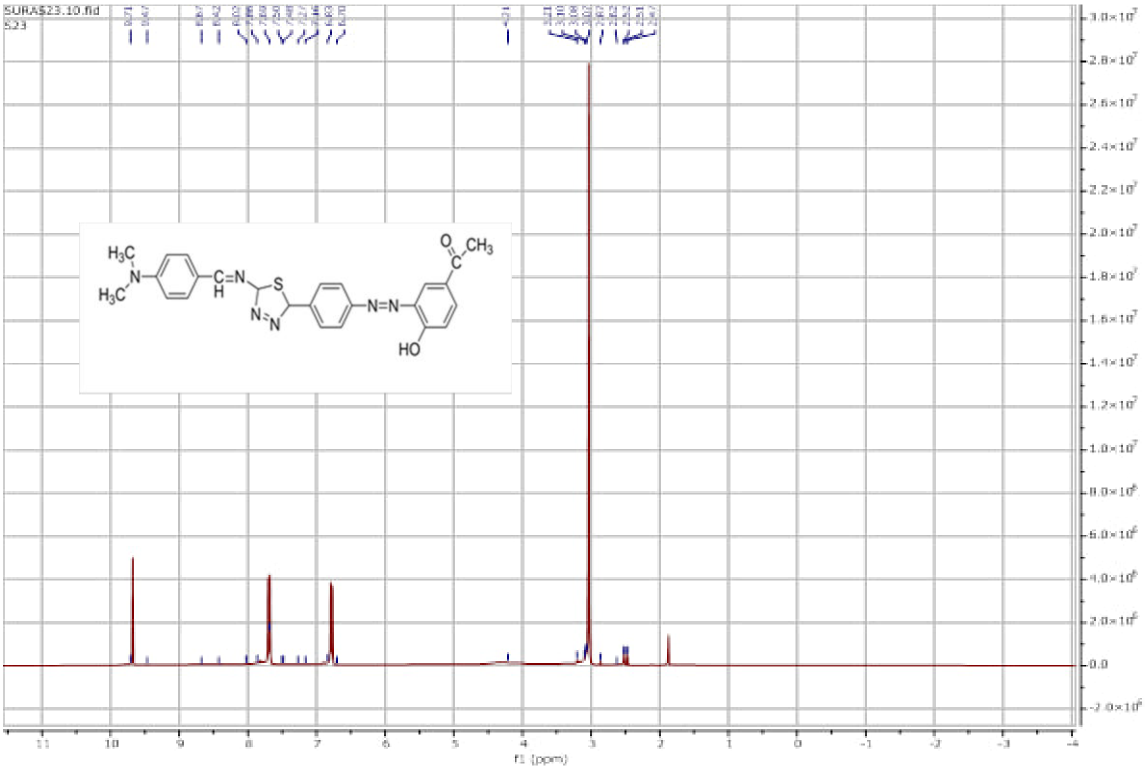
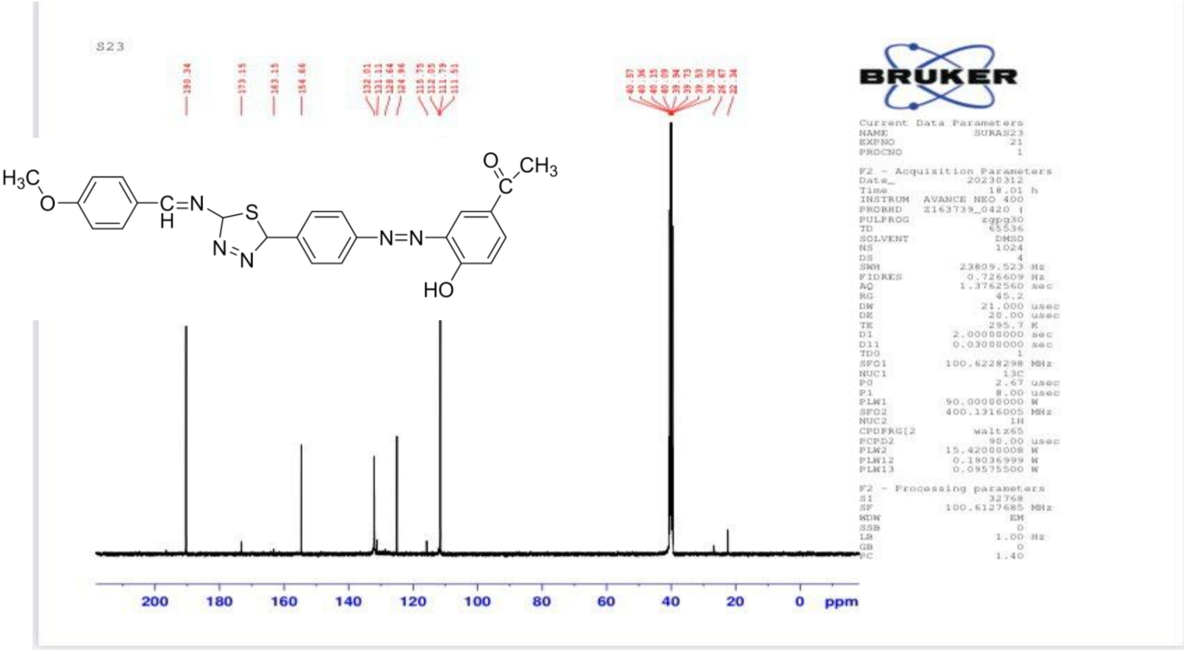


Figure (3-64) FT-IR Spectrum of compound[S23]



Figure(3-65) ^{1}HNMR of compound[S23]



Figure(3-66) ^{13}CNMR of compound[S23]

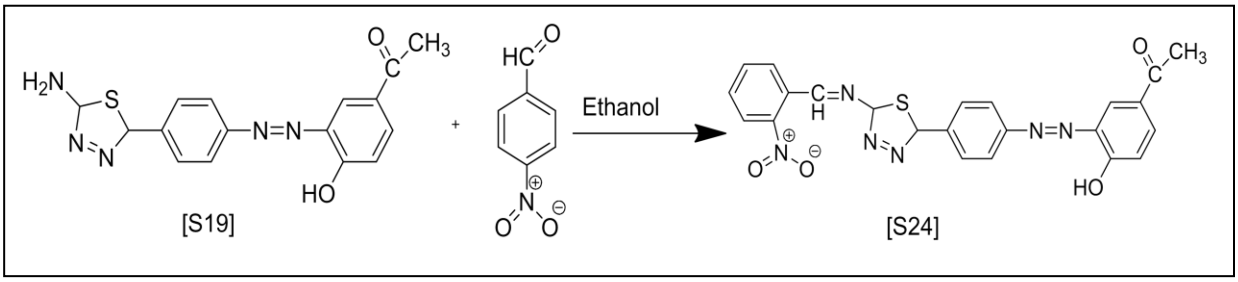
3.18 Synthesis of compound [S24]

Thiadiadiazole reacts with aldehyde(*o*-nitrobenzaldehyd) to form a Schiff base. According to the FT-IR technique, the amine group disappeared(3466-3327) and the C=N group appeared at(1680).

The FTIR(*v*, cm^{-1}): spectrum of compound [S24] exhibited absorption band at (1680) cm^{-1} for CH=N$_{\text{Schiff}}$,C-H Ar (3070), (1600) cm^{-1} for C=O $_{\text{ketone}}$, N=N (1541), C-H $_{\text{Alpha}}$ (2980) .

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 9.1 for N-H ,2.5 for C-H$_{\text{Alpha}}$, (11H,Ar-H) : 6.8-7.8 ,(C-H$_{\text{Endocyclic}}$) : 4.5 , (CH=N) :9.1.

^{13}C-NMR (100 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at 115-131.8 for (18C,Ar-C), and signal at 134.56 for CH=N$_{\text{Schiff}}$, (145.5) : C-OH , C-H$_{\text{Alpha}}$ (26.68).



Equation 3.19 synthesis of compound[S24]

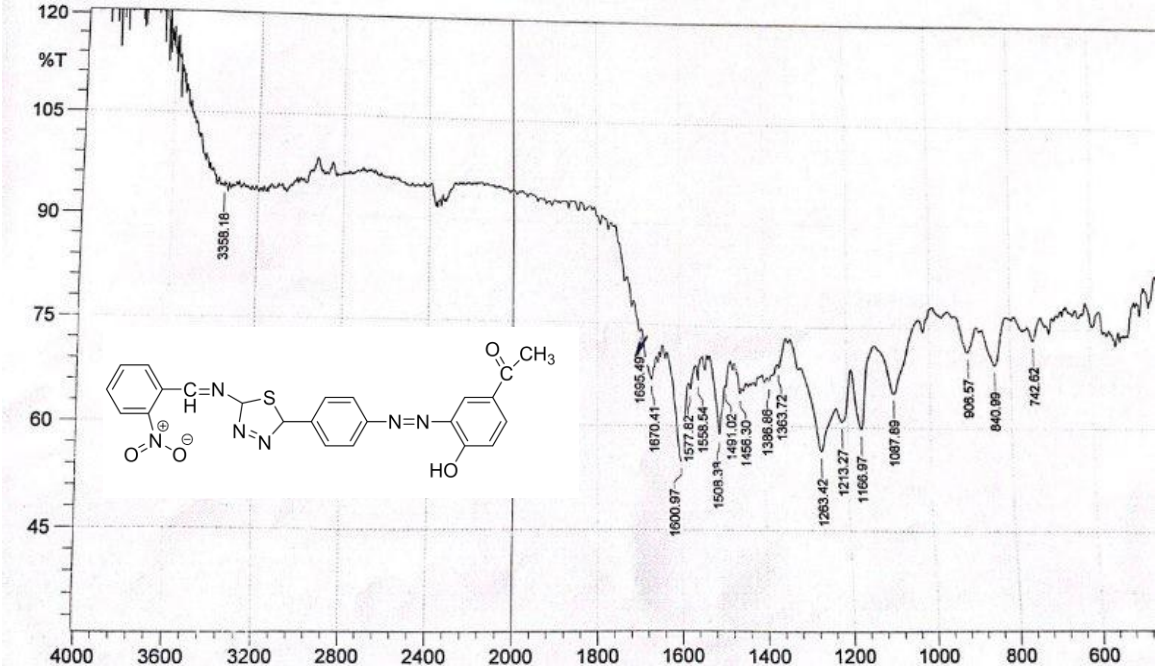
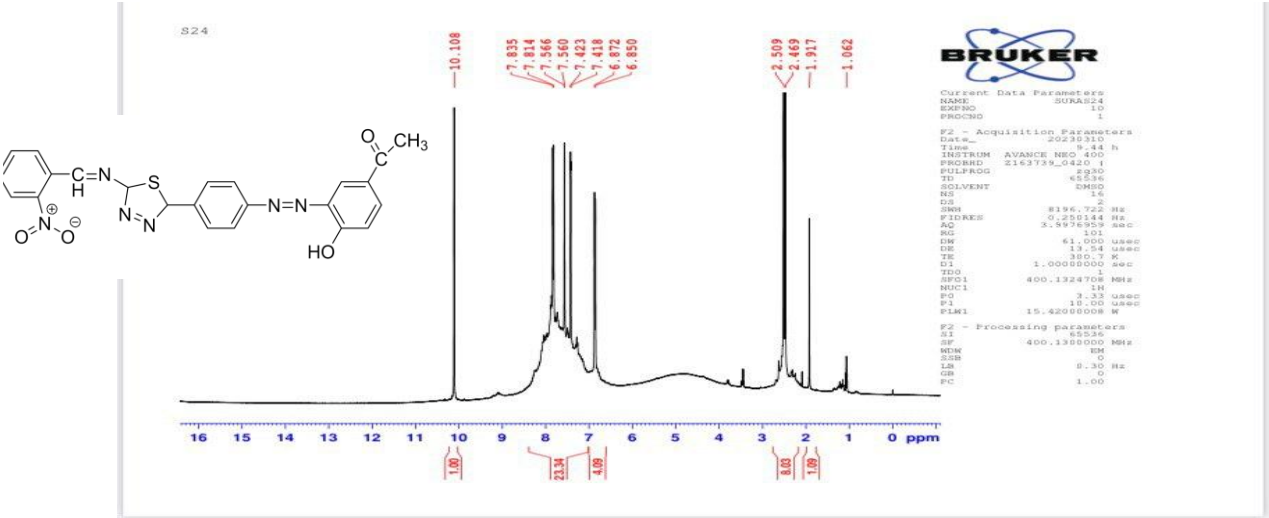
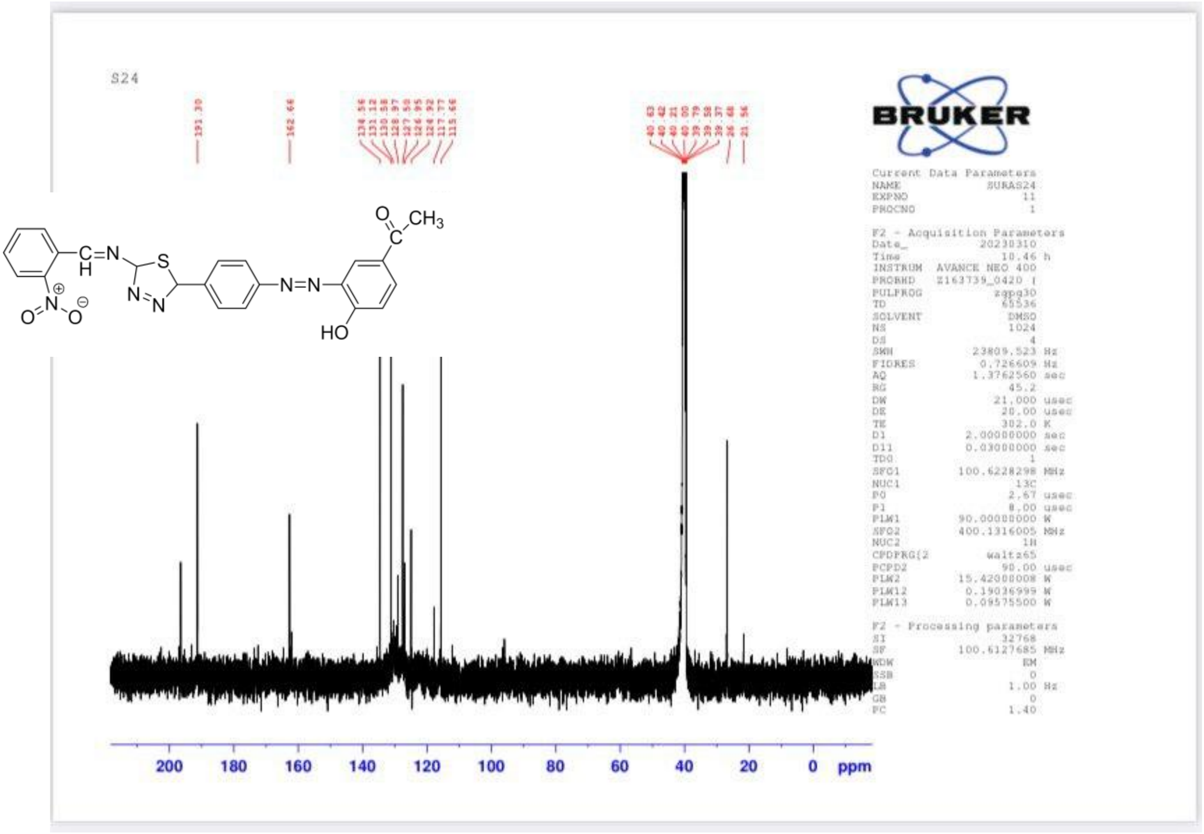


Figure (3-67) FT-IR Spectrum of compound[S24]



Figure(3-68) ^{1}HNMR of compound[S24]



Figure(3-69) ^{13}CNMR of compound[S24]

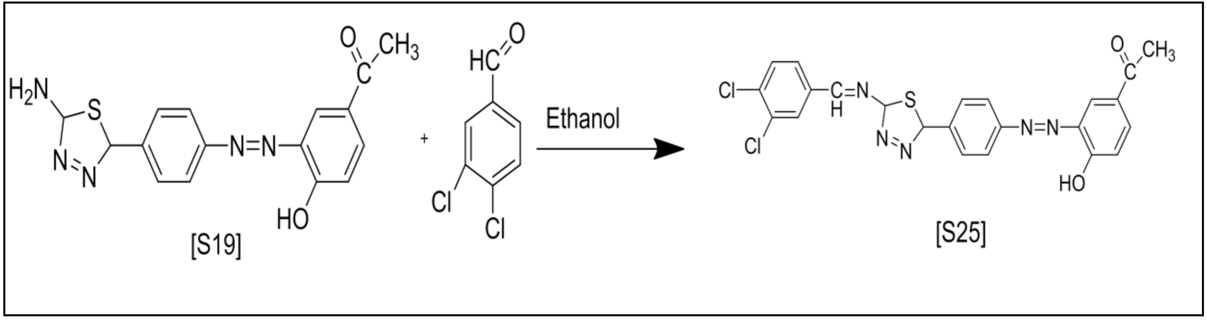
3.19 Synthesis of compound [S25]

Thiadiadiazole reacts with aldehyde(dichlorobenzaldehyd) to form a Schiff base. According to the FT-IR technique, the amine group disappeared(3466-3327) and the C=N group appeared at(1647),),(1602) for C=O$_{\text{keton,}}$ (1541) for N=N

The FTIR (v, cm^{-1}): spectrum of compound [S25] exhibited absorption band at (1647) cm^{-1} for C=N,(1602) cm^{-1} for C=O$_{\text{ketone}}$,O-H (3100),C-H $_{\text{alpha}}$ (2877), C=O $_{\text{keton}}$ (1602), N=N (1541), CH=N$_{\text{Schiff}}$ (1647) .

^{1}H-NMR(400MHz,DMSO-d6) :(δ, ppm) Spectrum showed appearance signal at 8.26 for CH=N$_{\text{Schiff}}$,2.51 for C-H$_{\text{Alpha}}$, (11H,Ar-H) : 6.3-7.9 ,(C-H$_{\text{Endocyclic}}$) : 3.9

^{13}C-NMR (400 MHz,DMSO-d6) :(δ, ppm) spectrum showed signals at 111-131 for (18C,Ar-C), and signal at 26.0 for, C-H $_{alpha}$, (135) : CH=N $_{Schiff}$, (190) :C=O $_{Ketone}$



Equation 3.20 synthesis of compound[S25]

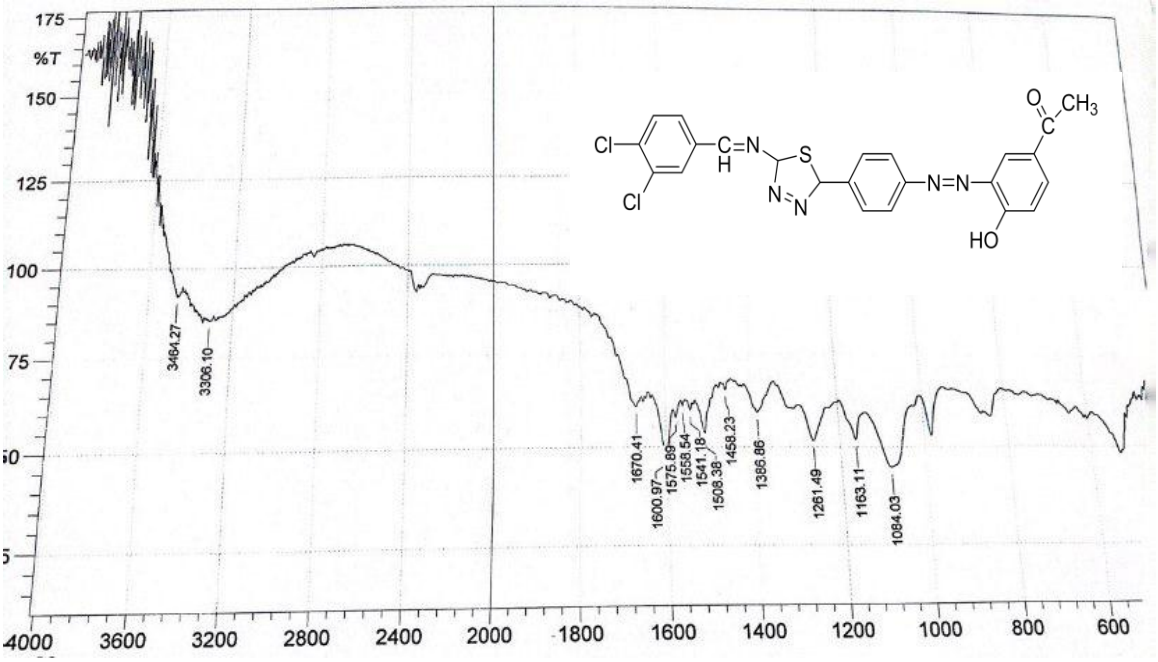
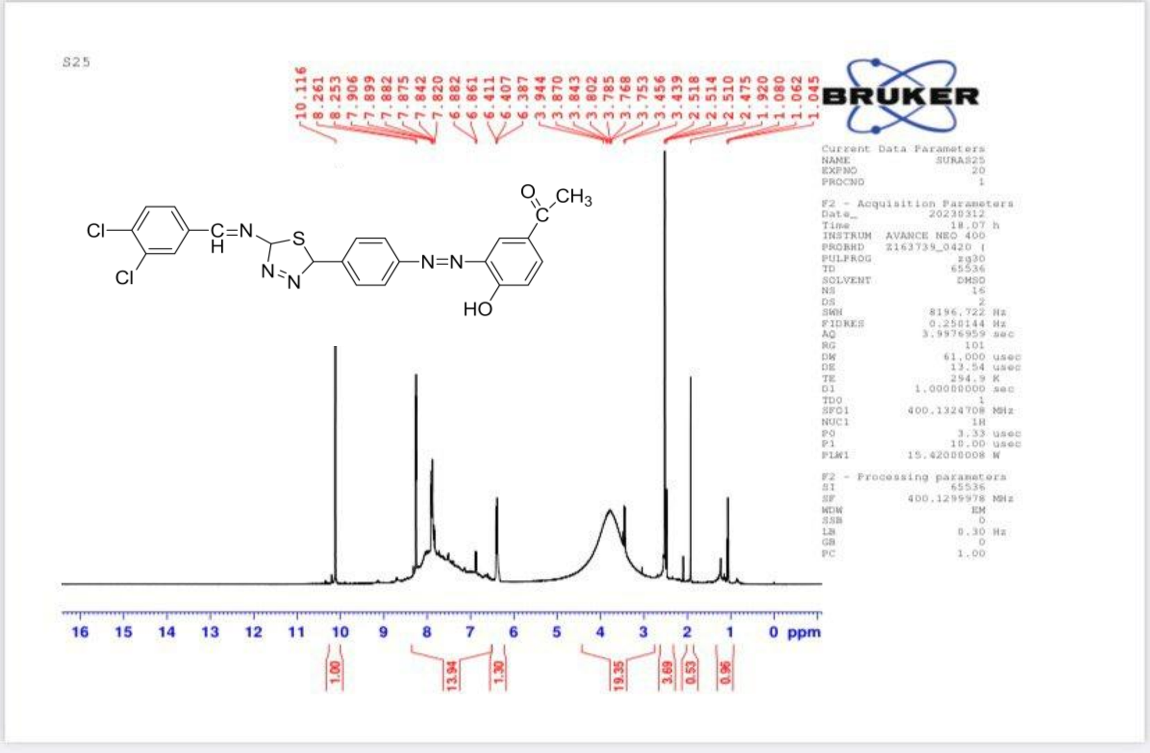
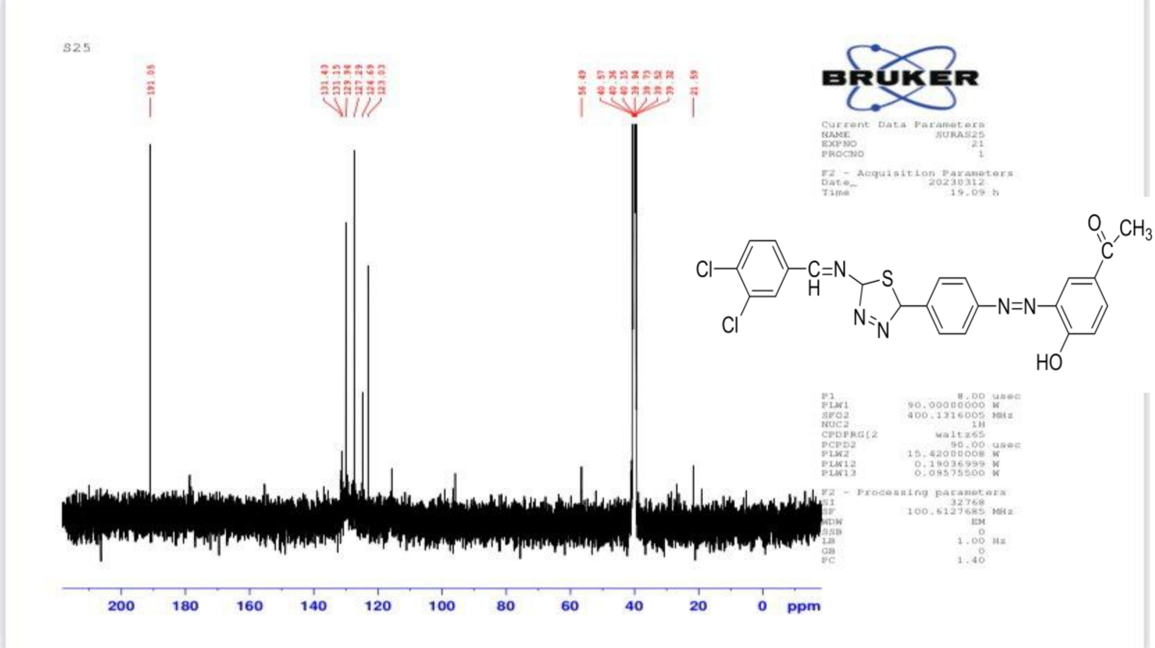


Figure (3-70) FT-IR Spectrum of compound[S25]



Figure(3-71) ^{1}HNMR of compound[S25]



Figure(3-72) ^{13}CNMR of compound[S25]

Table(3-1): C.H.N.S data of the prepared compounds [S1-S25]

Comp.No	C% Calculation	C% Measure	H% Calculation	H% Measure	N% Calculation	N% Measure	S% Calculation	S% Measure
[S1]	60.34	60	4.25	4.03	9.6	5.8	-	-
[S2]	66.32	65.8	4.5	4.01	13.63	13.2	-	-
[S3]	61.41	60.0	4.1	3.7	12.2	11.5	-	-
[S4]	59.7	58.7	4.3	3.6	17.6	17.06	6.5	5.9
[S5]	67.02	66.71	4.2	3.7	10.7	10	-	-
[S6]	66.7	65.9	4.5	3.9	8.6	8.07	-	-
[S7]	64.4	63.86	4.3	3.6	7.7	6.99	6.5	5.089
[S8]	62.4	61.8	3.8	3.06	12.5	11.91	-	-
[S9]	59.5	58.9	3.4	2.5	9.7	8.3	-	-
[S10]	63.6	62.84	3.8	3.03	9.9	9.0	-	-
[S11]	66.02	65.67	4.30	3.95	6.6	6.01	-	-
[S12]	62.8	62.06	3.93	3.03	12.22	11.9	-	-
[S13]	60.75	59.99	3.79	3.0	11.8	11.01	6.75	5.98
[S14]	68.50	67.8	4.33	3.79	11.02	10.69	-	-
[S15]	77.67	69.99	4.91	4.0	9.37	8.79	-	-
[S16]	66.41	65.85	4.198	3.47	8.01	7.87	6.106	5.87
[S17]	64.48	63.85	3.73	2.96	13.08	12.57	-	-
[S18]	64.33	63.87	3.49	2.69	9.79	9.05	-	-
[S19]	56.5	55.9	4.3	3.8	20.5	21.9	9.3	8.58
[S20]	61.6	60.98	4.4	3.9	14.7	14.05	6.7	5.9
[S21]	64.5	63.99	4.4	3.56	16.3	15.86	7.4	6.8
[S22]	62.2	61.8	4.5	3.95	15.45	14.98	7.6	7.02
[S23]	63.5	62.98	5.6	5.08	17.7	16.87	6.7	5.98
[S24]	58.2	57.6	3.7	3.01	17.7	16.8	6.6	6.04
[S25]	55.5	54.8	3.4	2.98	14.4	13.8	6.4	5.7

3.18 Biological Activity:

In this study, the activity of some types of bacteria *Staphylococcus epiderimidis*, *pseudomonas* (gram positive) and *Klipsiella* , *E. coli* (gram negative)were measured by an agar disc, where the compounds dissolved in dimethyl sulfoxide (DMSO) at a concentration of(10^{-3} M). It was observed that the compounds differed in their sensitivity according to the different types of bacteria. In the gram positive(*pseudomonas)* bacteria, compound [S4]appeared to be more effective than the rest of the compounds because it contained sulfur in its composition, while this effect was less with staph bacteria. While in the negative gram, the compound[S5] showed a little effect on (*E. coli)* bacteria, while the same compound showed a high effect on (*Klipsiella*) bacteria due to the presence of the nitrogen element in a greater amount. According to the results the compounds who had strong anti-bacterial action because of their structural contact with certain bacteria's cell walls, which resulted in high inhibition.

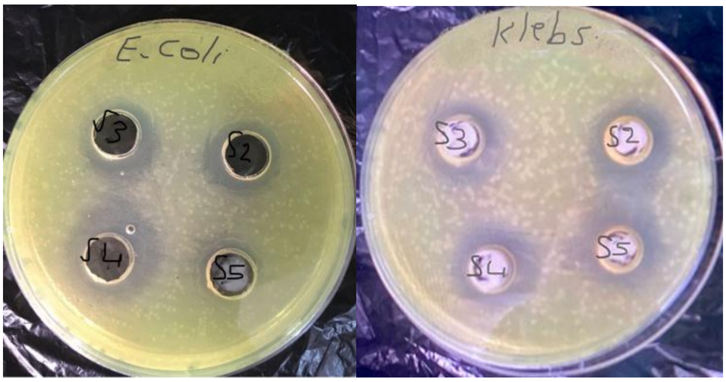


Image (3-1) :Effect of compounds[S2-S5] on *Staphylococcus Epiderimidis*, *pseudomonas*, *Klebsiella pneumoniae, and E.Coli.*

Table(3-2) antibacterial activity of the compounds[S2-S5]

No. of com	*Staphylococcus Epiderimidis*	*pseudomonas*	*Klebsiella pneumoniae*	*E.Coli*
S2	27	22	25	23
S3	26	22	19	23
S4	9	33	26	30
S5	25	23	25	11

3.19 Corrosion Measurement:

The corrosion inhibition of prepared compounds[S11,S13,S18] Have been studied on Carbone Steele surface area in (0.1) M hydrochloric acid solution at different temperature (293-303-313)K. polarization method was used to evaluate the inhibition efficiency of the above compounds, IE% was calculated in the equation below:

$$\mathbf{\%IE = \frac{(icorr)o-(icorr)}{(icorr)o} * 100}$$

Where $(i_{corr})_o$is the corrosion current density in the absence of inhibitors, (i_{corr}) is the corrosion current density in the presence of inhibitors

Table 3.2 showed good inhibition efficiency for the synthesis compounds at different temperatures ,that give a maximum inhibition efficiency IE% result indicate that ring substitution in the designed compounds has a significant effect on the corrosion inhibition. It has been suggested that heteroatoms in the compound nucleus, polar functional groups, and conjugated double bonds as part of substitution have a key influence in the effectiveness of the inhibition**.**

Table (3-3) Corrosion parameters for blank and compound in HCl solutions and different compound.

Comp.	Temp.	E corr.	I corr.	I corr./ r	Resis.	Anodic β	Cathodic β	Corr. rate,	IE%
Blank	**293**	-0.430	248.9	2.489E-4	114.5	0.100	0.189	1.222	-
	303	-0.404	257.8	2.578E-4	89.47	0.192	0.073	1.265	-
	313	-0.382	275.1	2.751E-4	97.71	0.158	0.102	1.350	-
S11	**293**	-0.565	11.79	2.358E-5	3838	0.177	0.253	0.116	95
	303	-0.601	13.57	2.713E-5	4965	0.287	0.337	0.133	95
	313	-0.627	15.28	3.055E-5	4677	0.345	0.315	0.150	94
S13	**293**	-0.568	17.17	3.434E-5	5251	0.402	0.430	0.169	93
	303	-0.481	18.86	3.771E-5	3947	0.243	0.581	0.185	93
	313	-0.626	20.50	4.099E-5	4119	0.482	0.326	0.201	93
S18	**293**	-0.590	22.47	4.494E-5	3338	0.325	0.369	0.221	91
	303	-0.609	23.56	4.711E-5	3486	0.384	0.373	0.231	91
	313	-0.637	23.92	4.783E-5	3120	0.396	0.303	0.235	91

E corrosion, V

I corrosion, μA

I corrosion per surface area, A/cm²

Polarization Resistance, Ω

Anodic β Tafel constant, V/decade

Cathodic β Tafel constant, V/decade

Corrosion rate, mm/year

IE% inhibition efficiency

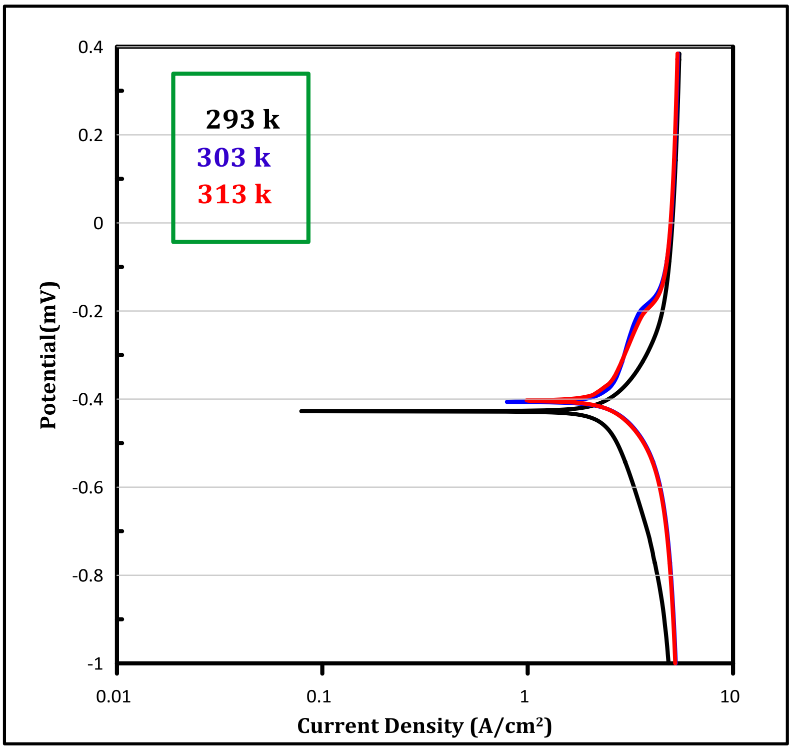


Figure 3-74 Polarization curves for corrosion of blank HCl solution.

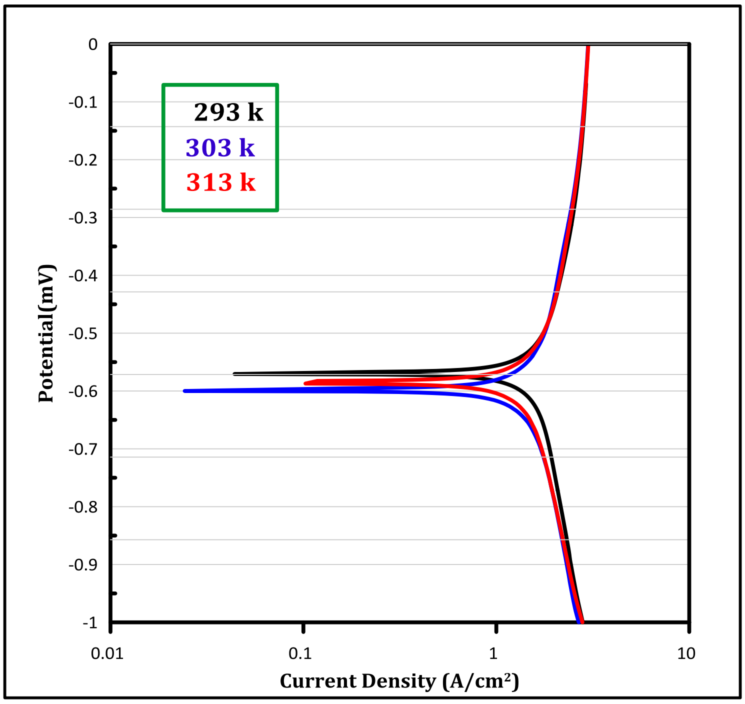


Figure 3-75 Polarization curves for corrosion of [S11].

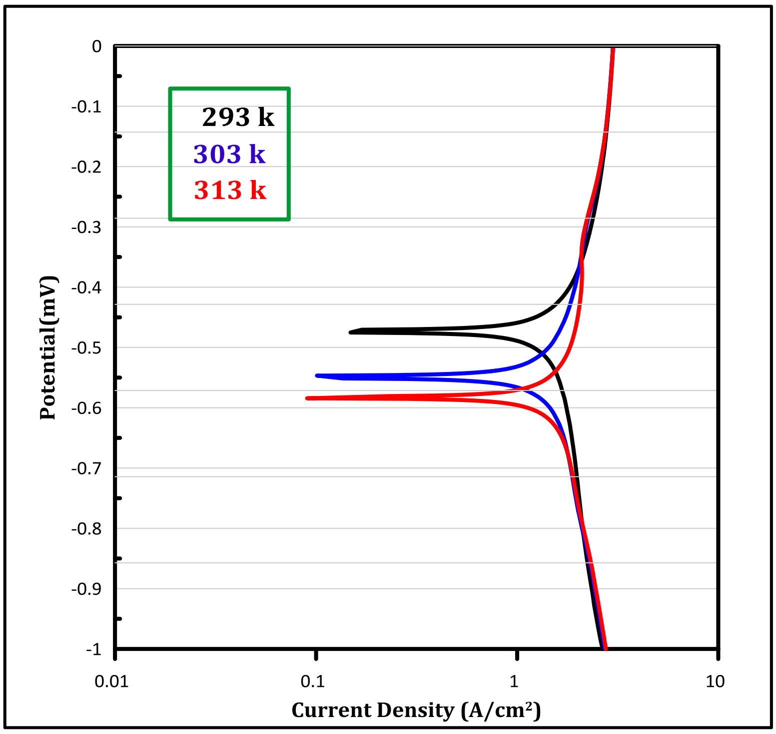


Figure 3-76 Polarization curves for corrosion of [S13].

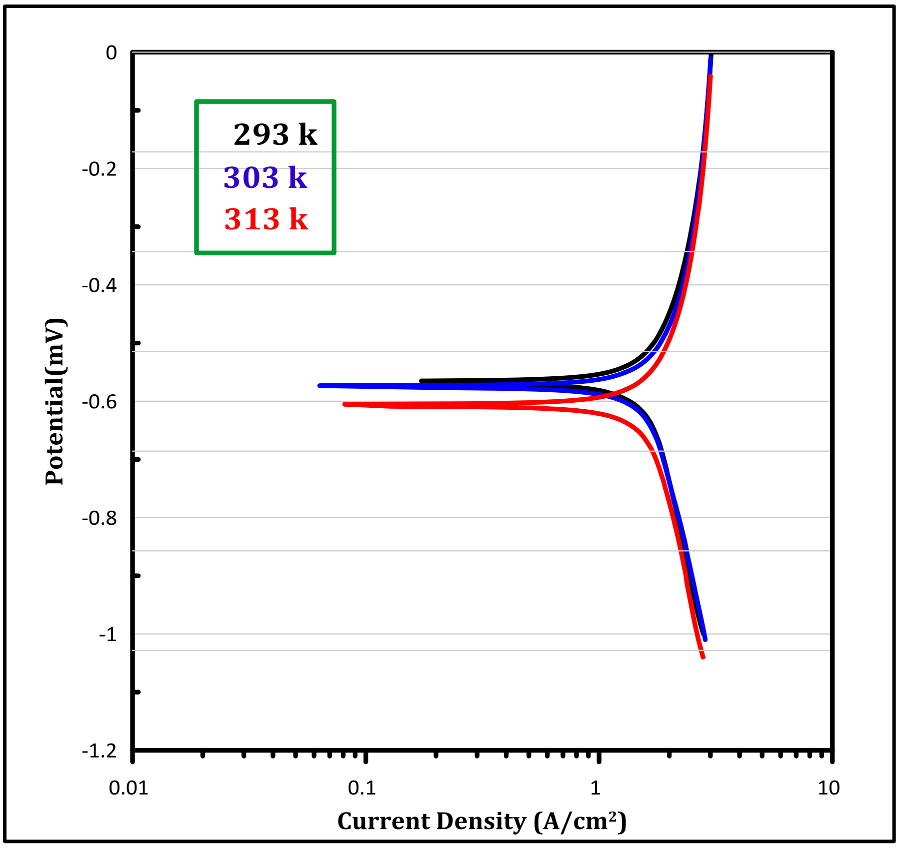
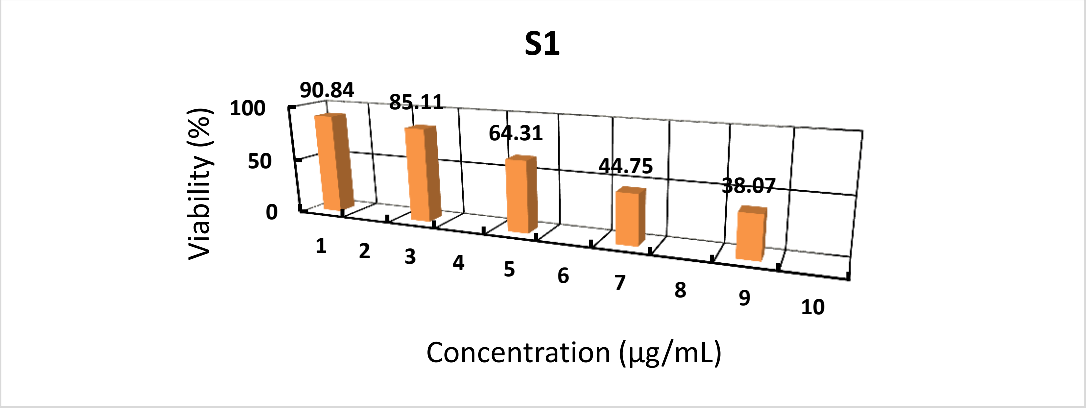


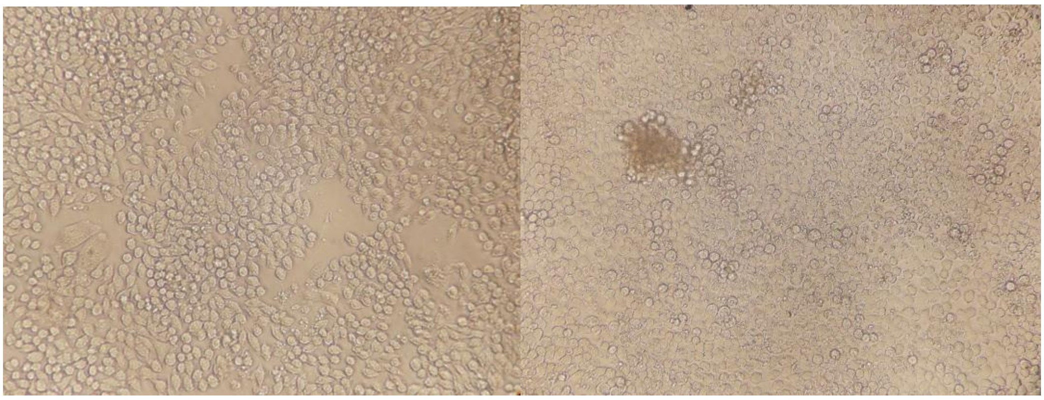
Figure 3-78 Polarization curves for corrosion of [S18].

3.20 Anti-Cancer Measurements:

In this study, the breast cancer cell line (mcf7) and the healthy cell line (mcf10) were used for comparison and to find out the extent of their effectiveness on human body cells and the possibility of using them as cancer drugs. Then the test (MTT) was used for the biological examination of all cells. When studying the effect of compounds (S1,S17,S19) On the growth process of cancerous cell lines of breast cancer (mcf7) and normal healthy cells (mcf10) it was observed that the compound [S17] has value of IC50 equal to(237.5$_{mg/ml}$) and was for compound [S19] (196.6$_{mg/ml}$) ,So that the [S17,S19] have low effect on the breast cancer than [S1] (37.5$_{mg/ml}$) . While on the normal healthy cells (mcf10), the value of IC50 for compound [S1] equal to (200.3$_{mg/ml}$) and for [S17,S19] equal to (601.4$_{mg/ml}$,675.0$_{mg/ml}$) respectively so that lead to know these two compounds have high effect on the cells.



Effective of compound [S1] on normal breast cells

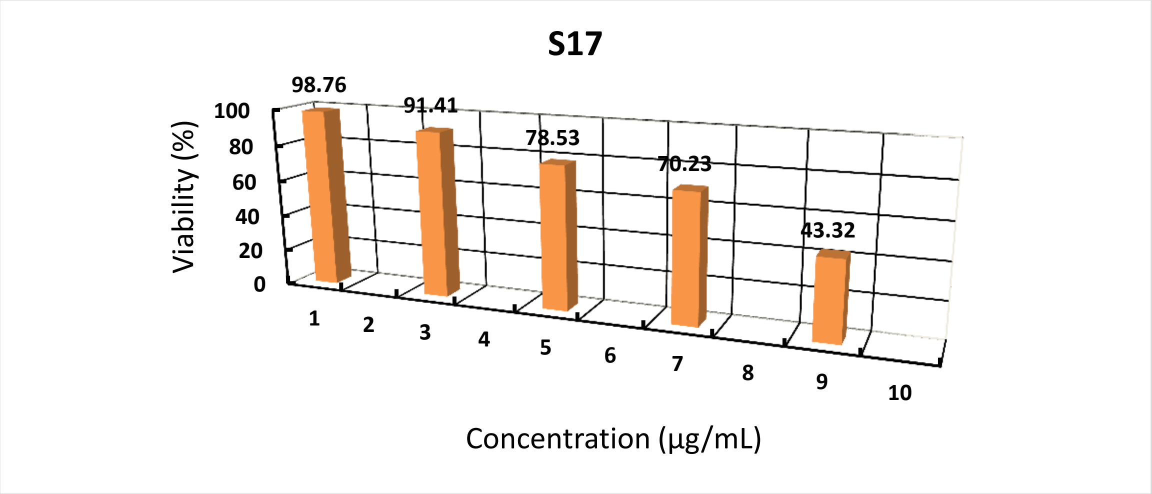


Image(3-2) compound [S1] on normal breast cells

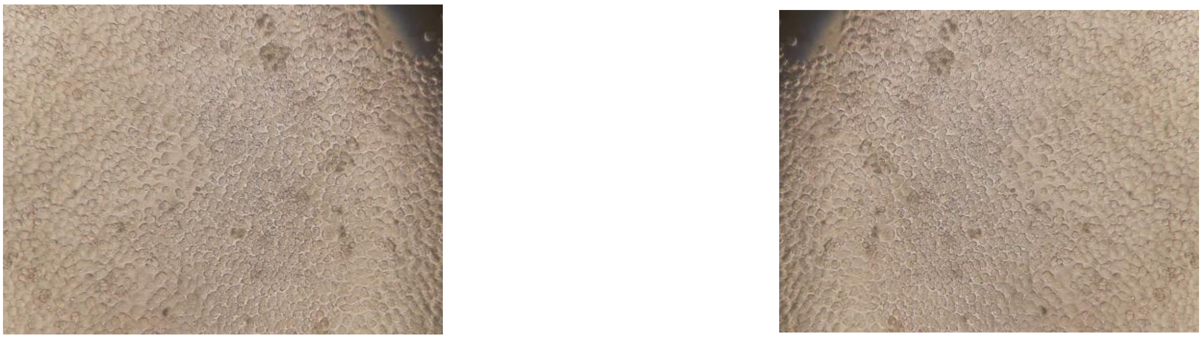
Table(3-4) The anti-cancer cultures of compound [S1]on normal breast cancer cells

Sample ID	S1									
Concentration (µg/mL)	7.4		22.22		66.66		200		600	
absorption at 570 nm	0.498	0.454	0.401	0.491	0.333	0.341	0.213	0.256	0.200	0.199
Viability (%)	95.04	86.64	76.53	93.70	63.55	65.08	40.65	48.85	38.17	37.98
Average Viability (%)	90.84		85.11		64.31		44.75		38.07	
Standard Deviation (±)	5.94		12.14		1.08		5.80		0.13	

IC50	**200.3 µg/mL**



Effective of compound [S17] on normal breast cells

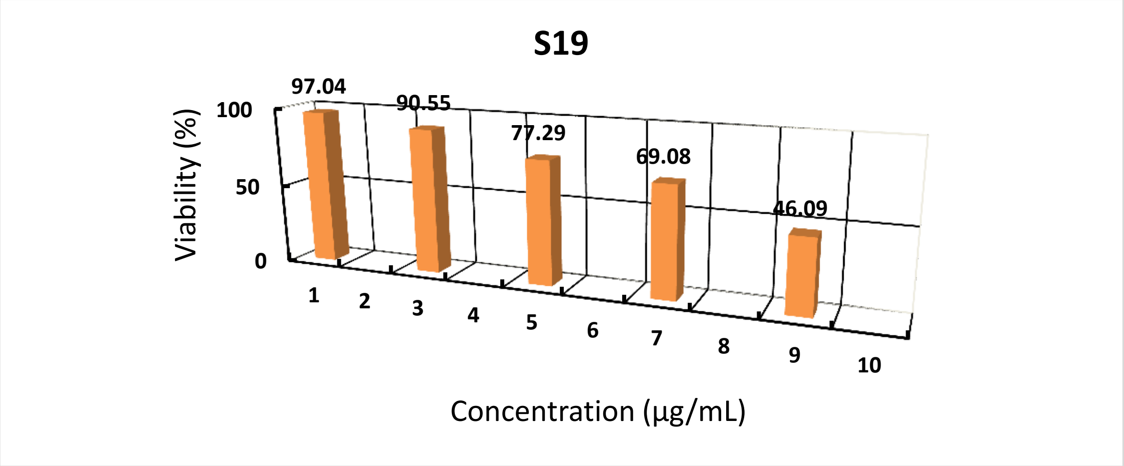


Image(3-3) compound [S17] on normal breast cells

Table(3-5) The anti-cancer cultures of compound [S17]on normal breast cancer cells

Sample ID	S17									
Concentration (µg/mL)	7.4		22.22		66.66		200		600	
absorption at 570 nm	0.522	0.513	0.477	0.481	0.401	0.422	0.380	0.356	0.211	0.243
Viability (%)	99.62	97.90	91.03	91.79	76.53	80.53	72.52	67.94	40.27	46.37
Average Viability (%)	98.76		91.41		78.53		70.23		43.32	
Standard Deviation (±)	1.21		0.54		2.83		3.24		4.32	

IC50	**601.4**	**µg/mL**



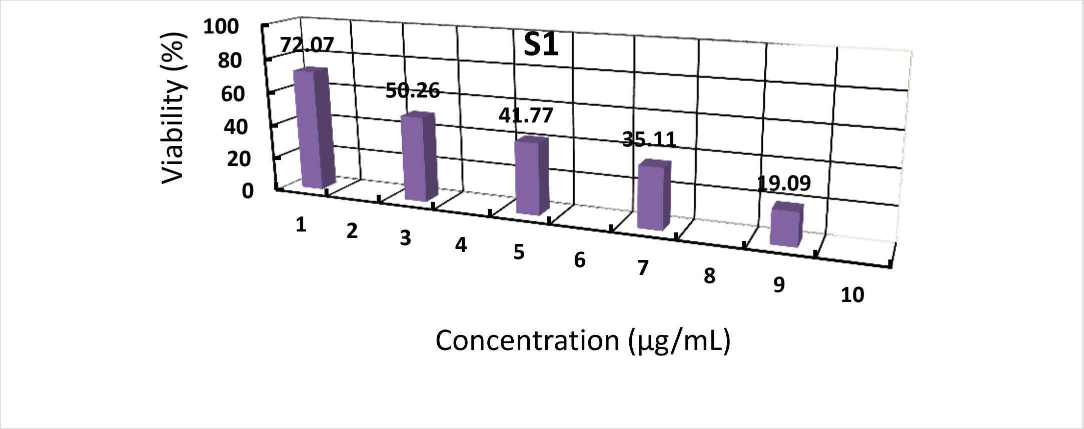
Effect of compound [S19] on normal breast cancer cells

Image(3-4) compound [S17] on normal breast cells

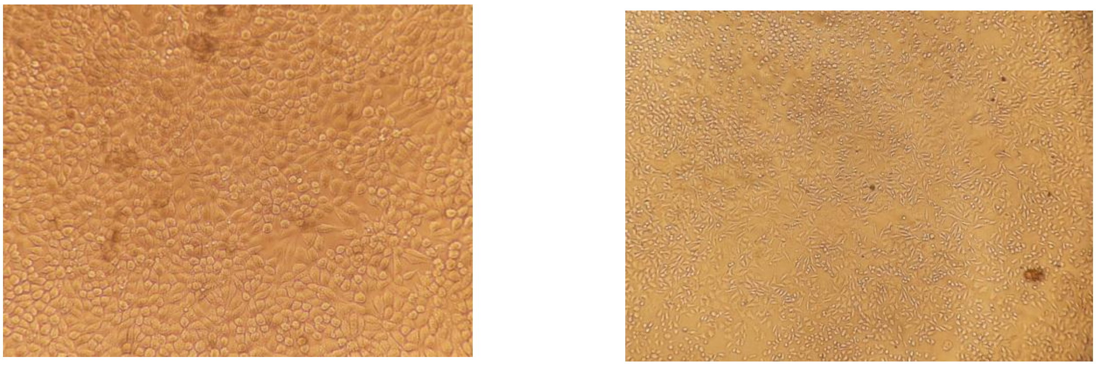
Table(3-6) The anti-cancer cultures of compound [S19]on normal breast cancer

Sample ID	S19									
Concentration (µg/mL)	7.4		22.22		66.66		200		600	
absorption at 570 nm	0.516	0.501	0.483	0.466	0.421	0.389	0.351	0.373	0.233	0.250
Viability (%)	98.47	95.61	92.18	88.93	80.34	74.24	66.98	71.18	44.47	47.71
Average Viability (%)	97.04		90.55		77.29		69.08		46.09	
Standard Deviation (±)	2.02		2.29		4.32		2.97		2.29	

IC50	**675.0 µg/mL**



Effect of compound [S1] on breast cancer cells

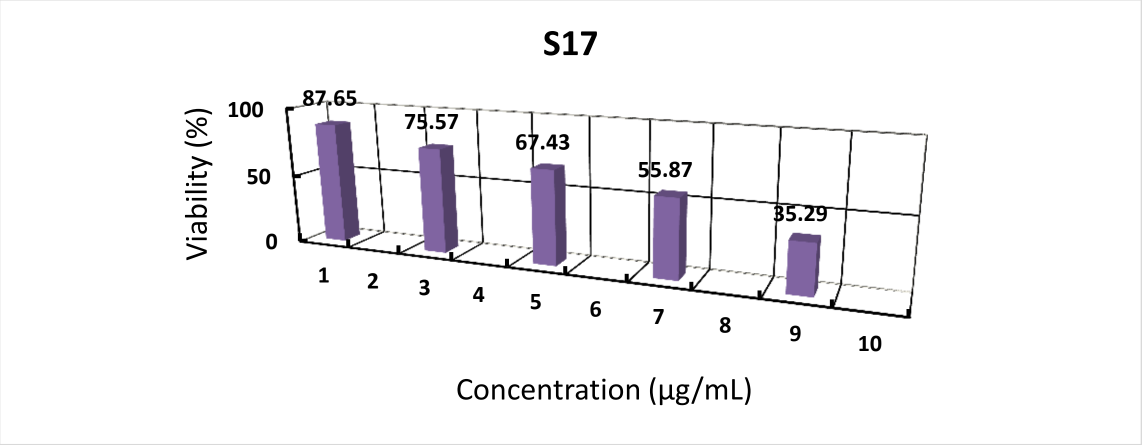


Image(3-5) compound [S1] on breast cancer cells

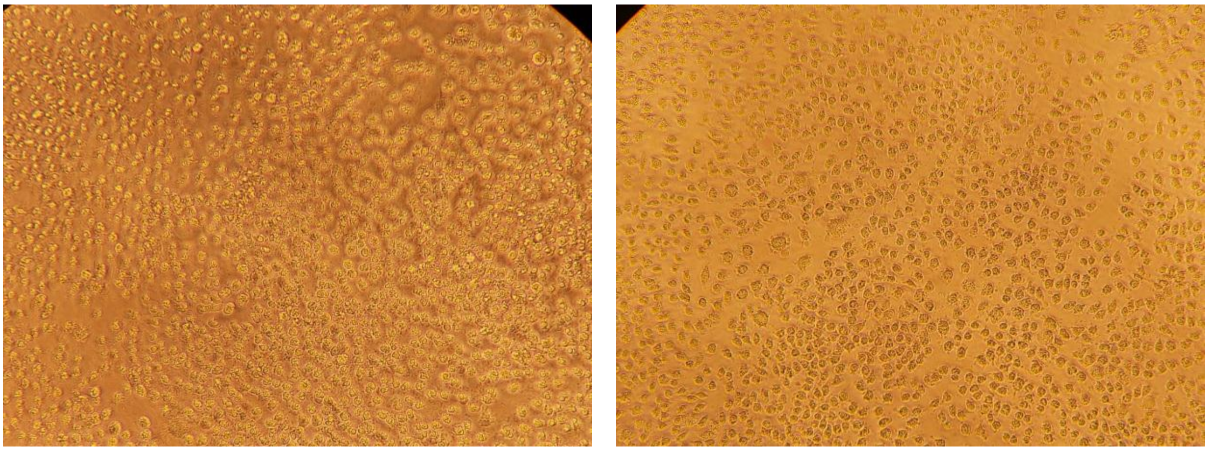
Table(3-7) The anti-cancer cultures of compound [S1]on breast cancer cells

Sample ID	S1									
Concentration (µg/mL)	7.4		22.22		66.66		200		600	
absorption at 570 nm	0.396	0.427	0.281	0.293	0.240	0.237	0.195	0.206	0.105	0.113
Viability (%)	69.35	74.78	49.21	51.31	42.03	41.51	34.15	36.08	18.39	19.79
Average Viability (%)	72.07		50.26		41.77		35.11		19.09	
Standard Deviation (±)	3.84		1.49		0.37		1.36		0.99	

IC50	**37.5 µg/mL**



Effect of compound [S17] on breast cancer cells

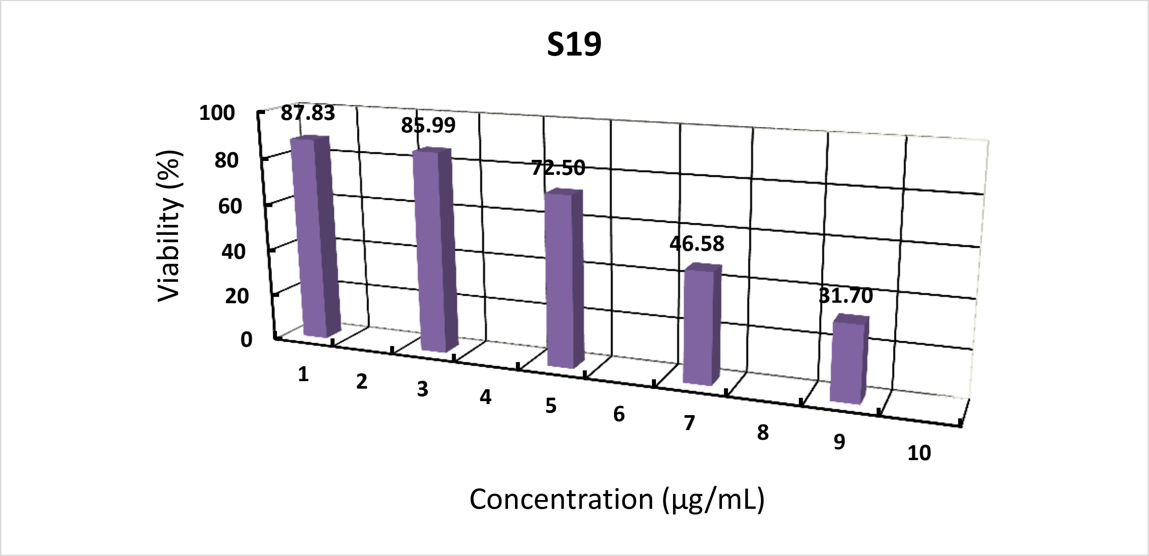


Image(3-6) compound [S17] on breast cancer cells

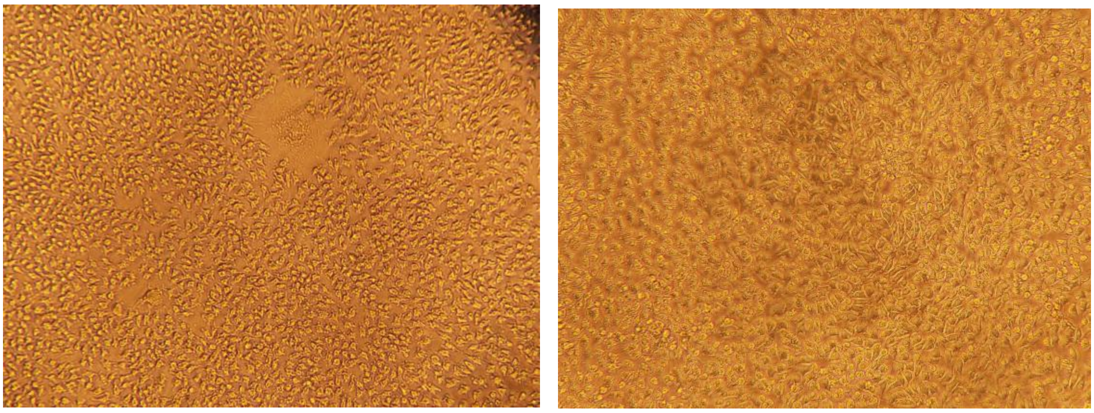
Table(3-8) The anti-cancer cultures of compound [S17]on breast cancer cells

Sample ID	S17									
Concentration (µg/mL)	7.4		22.22		66.66		200		600	
absorption at 570 nm	0.511	0.490	0.458	0.405	0.356	0.414	0.368	0.270	0.185	0.218
Viability (%)	89.49	85.81	80.21	70.93	62.35	72.50	64.45	47.29	32.40	38.18
Average Viability (%)	87.65		75.57		67.43		55.87		35.29	
Standard Deviation (±)	2.60		6.56		7.18		12.14		4.09	

IC50	**237.5 µg/mL**



Effect of compound [S19] on breast cancer cells



Image(3-7) compound [S19] on breast cancer cells

Table(3-9) The anti-cancer cultures of compound [S19]on breast cancer cells

Sample ID	S19									
Concentration (µg/mL)	7.4		22.22		66.66		200		600	
absorption at 570 nm	0.536	0.467	0.501	0.481	0.439	0.389	0.266	0.266	0.159	0.203
Viability (%)	93.87	81.79	87.74	84.24	76.88	68.13	46.58	46.58	27.85	35.55
Average Viability (%)	87.83		85.99		72.50		46.58		31.70	
Standard Deviation (±)	8.54		2.48		6.19		0.00		5.45	

IC50	**196.6 µg/mL**

Table (3-10) Acomparision between IC_{50} values of compounds [S1,S17,S19] on normal breast cells and cancer breast cells.

Synthesis compounds		IC_{50} values (normal cell)	IC_{50} values (cancer cell)
S1	4-((5-acetyl-2,4-dihydroxyphenyl)diazenyl) benzoic acid	200.3	37.5
S17	-2))-hydroxy-5-(5-(4-hydroxy-3-methoxyphenyl)-4,5-dihydro-1H-pyrazol-3-yl)phenyl)diazenyl)benzoic acid	601.4	237.5
S19	-5)-4))-3)-amino-2,5-dihydro-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)-4-hydroxyphenyl)ethanone	675.0	196.6

3.21 Conclusions:

1. synthesis of azo dye ,chalcon with a high yield .

2. Several new heterocyclic compounds were synthesized from chalcon with very good yield.

3.Several new Schiff- base heterocyclic compounds were synthesized from thiadiazole amine with very good yield and confirmed by FT-IR,^{1}HNMR and ^{13}CNMR.

4. The physical properties of these compounds have been investigated.

5.Study the biological activity of the (five and six)membered heterocyclic compounds and were achieved excellent results, Due to the presence of sulfur in its chemical makeup, compound [S4] was found to be more effective against bacteria than its peers.

6. The corrosion resistance of some compounds[S11,S13,S18] has been studied and it was found that the tested compounds are good anti-corrosion.

7.The anti-cancer test for some compounds[S1,S17,S19] show a high effect on the breast cancer cell ,so it is represented a good anti-cancer.

3.22. Suggestion For Future Work:

1.Synthesis of new five, six and seven heterocyclic compounds from chalcon.

2. Synthesis of imidazole derivatives from chalcon.

3.Synthesis of new Schiff base from the thiadiazole amine .

4. Synthesis of new Azo compounds from the thiadiazole amine .

5. Synthesis of Chalcone Imine from Chalcone compounds

6. Study the anti-cancer of the prepared compounds.

7. Study the anti-corrosion of the prepared compounds.

8.Study the anti-oxidant of the prepared compounds.

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الخلاصة:

تتضمن هذه الدراسة تخليق مركبات حلقية غير متجانسة جديدة تستخدم حامض أمينوبنزويك ونوعين من الكيتون كمواد أولية لإنتاج نوعين من مشتقات الجالكون والتي تستخدم لتخليق مركبات حلقية غير متجانسة جديدة مختلفة متكونة من خمسة ، ستة وسبعة حلقات غير متجانسة [S٣-S٩، S١٢-S١٨] وايضا تخليق مركبات حلقية غير متجانسة جديدة او ماتسمى بقواعد شف من تفاعل ١،٣،٤-ثياديازول أمين [S١٩] مع الثايوسيميكاربازايد، تم تمييز المواد العضوية المحضرة باستخدام تقنيات مختلفة: التحليل الطيفي FT-IR و ^{١}H-NMR و ^{١٣}C-NMR بالإضافة إلى تحليل CHNS.

ينقسم هذا العمل إلى أربعة أجزاء مختلفة:

الجزء الاول:

تحضير صبغة الآزو الجديدة [S١] من التفاعل بين حامض أمينو بنزويك و ٢،٤-ثنائي هيدروكسي أسيتوفينون ، يتفاعل هذا الآزو مع الألديهايد الاروماتي (٣-هيدروكسي-٤-ميثوكسي بنزالديهايد) لإنتاج مشتقات الجالكون [S٢]. يتفاعل هذا الجالكون مع مركبات مختلفة لإنشاء مركبات حلقية غير متجانسة جديدة ، كما يظهر في المخطط (١)

الجزء الثاني:

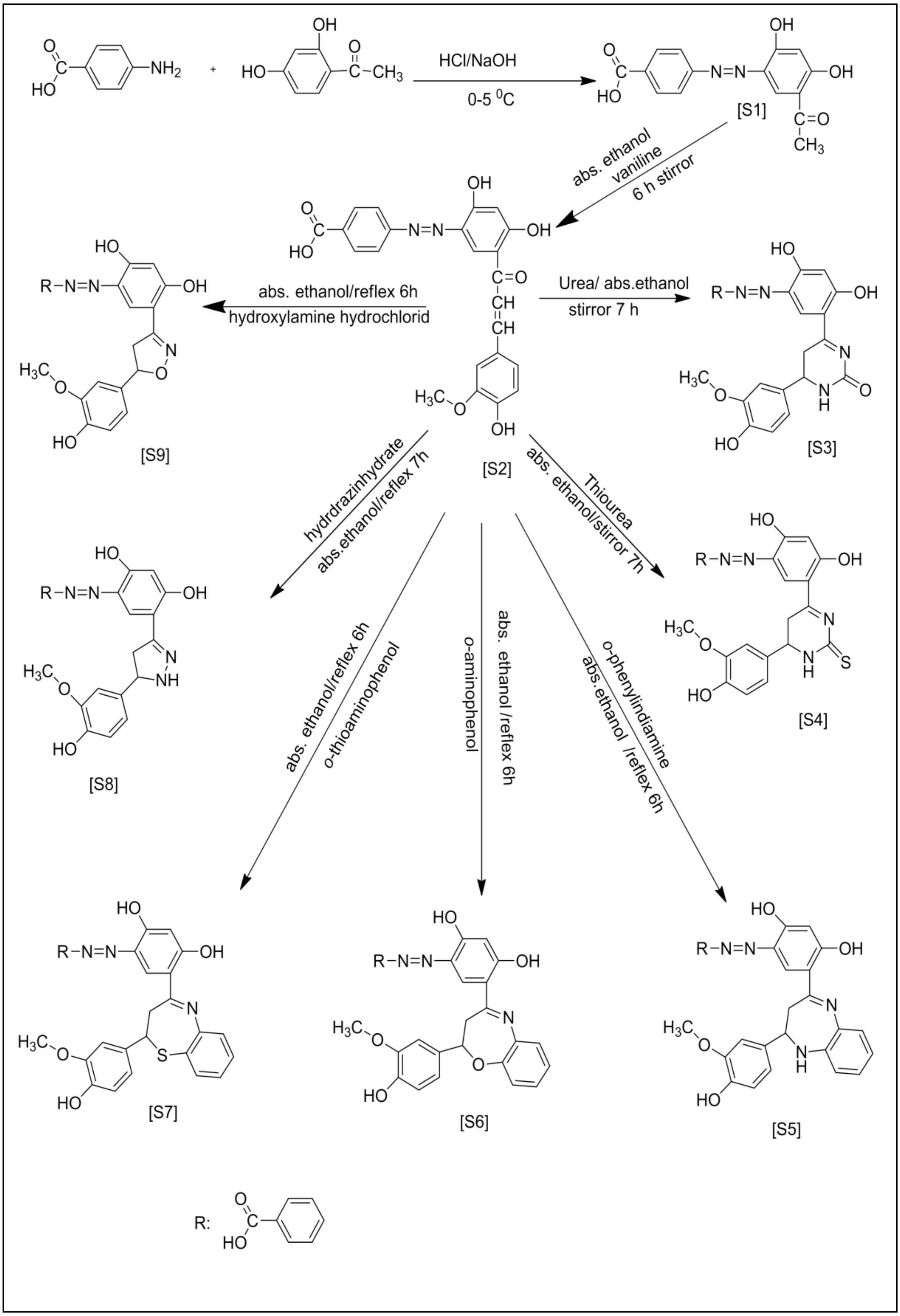
يتكون مركب الأزو الجديد [S١٠] من التفاعل بين حمض أمينو بنزويك و ٤-هيدروكسي أسيتوفينون ، ثم تتفاعل هذه الصبغة مع واحد من مشتقات الألديهيد (٣-هيدروكسي-٤-ميثوكسي بنزالديهايد) لإنتاج جالكون جديد [S١١] عن طريق تكاثف كلايسين شيميدت ، يتفاعل هذا الجالكون مع مركبات مختلفة لتخليق مركبات حلقية غير متجانسة [S١٢-S١٨] مخطط (٢) تظهر كل هذه التفاعلات.

الجزء الثالث:

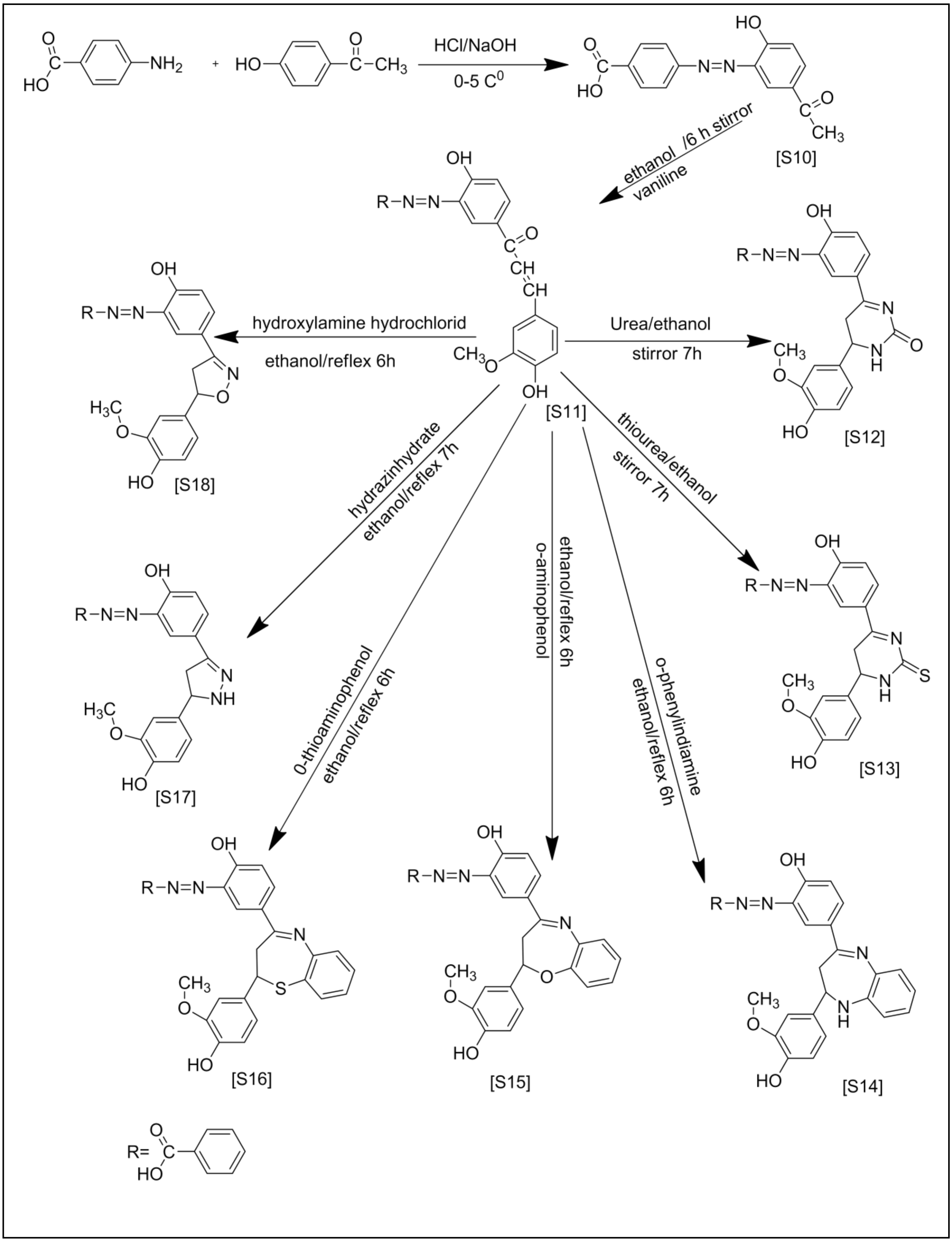
يتضمن هذا الجزء تحضير ٤,٣,١- ثاياديازول أمين [S١٩] عن طريق التفاعل بين [S١٠] وثايوسيميكاربازايد في وجود كلوريد الفسفور الثلاثي ، يتفاعل هذا الأمين مع ألديهايدات مختلفة لتوليد قواعد شيف جديدة [S٢٠-S٢٥] . مخطط (٣) يظهر جميع هذه التفاعلات.

الجزء الرابع:

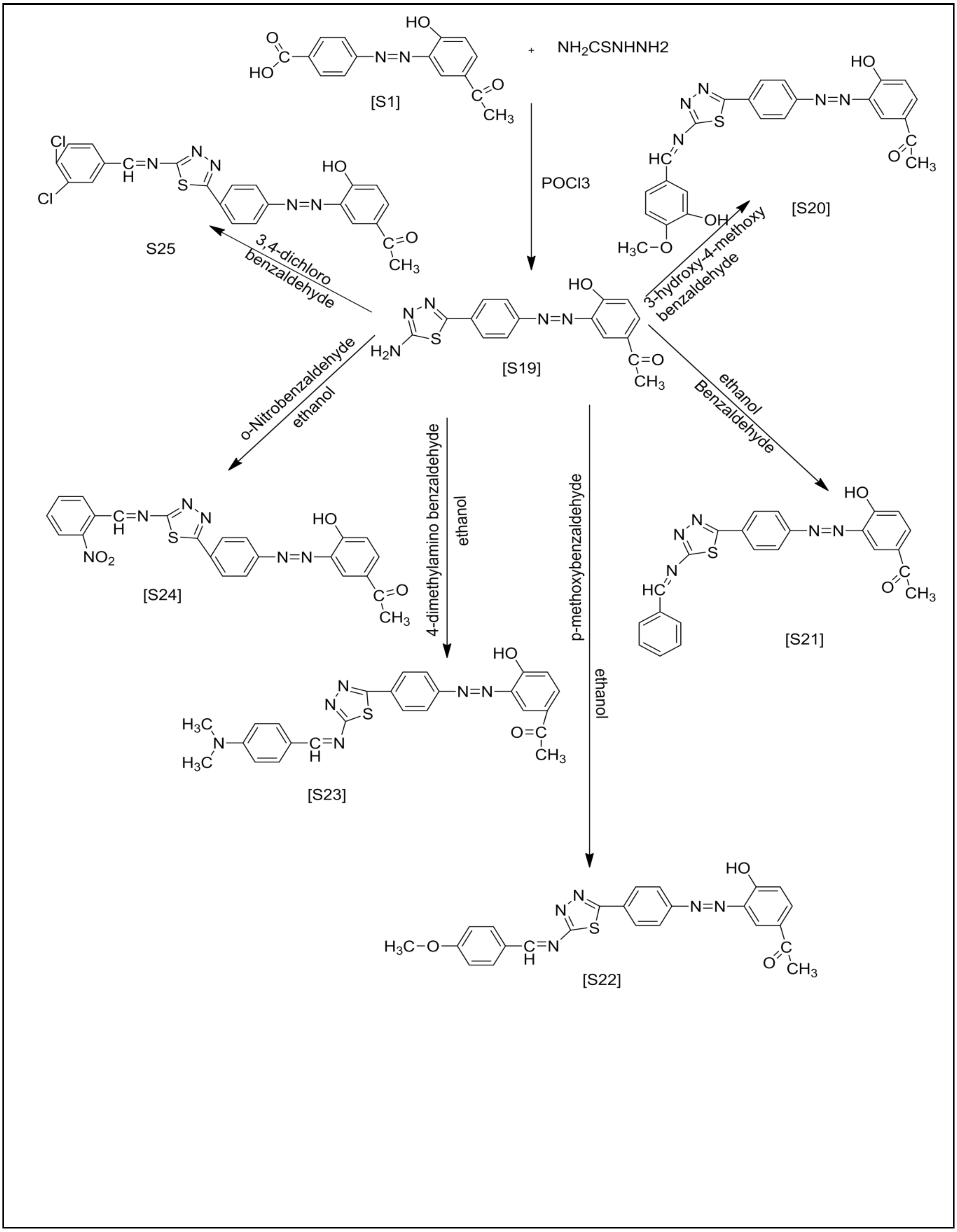
في هذا الجزء تمت دراسة النشاط البيولوجي لبعض أنواع المركبات المحضرة ضد أربعة أنواع من البكتيريا ، اثنتان منها موجبة الغرام (*Staphylococcus Epiderimidis*، *pseudomonas*) واثنان منها سالبة الغرام (*Klebsiella pneumonia*، *E.Coli*) ، وكذلك دراسة مضادة للتآكل ومضادة للسرطان لبعض أنواع المركبات.



مخطط (١) تحضير المركبات [S١-S٩]



مخطط (٢) تحضير المركبات [S١٠-S١٨]



مخطط (٣) تحضير المركبات [S١٩-S٢٥]

جمهورية العراق

وزارة التعليم العلي والبحث العلمي

جامعة بابل

كلية العلوم للبنات

تخليق وتشخيص المركبات الحلقية غير المتجانسة الجديدة أعتماداً على مشتقات بارا- أمينو حامض البنزويك ودراسة بعض تطبيقاتها

رسالة

مقدمة الى مجلس كلية العلوم للبنات – جامعة بابل

جزء من متطلبات الحصول على درجة ماجستير في العلوم/الكيمياء

سرى صادق عبيد السلطاني

بكالوريوس جامعة بابل ٢٠١٠

اشراف

أ.م.د. نور عبد الرزاق عبد اللطيف

١٤٤٤ه ٢٠٢٣م