Republic of Iraq
Ministry of Higher Education
and Scientific Research
University of Babylon
College of Science for Girls
Department of Chemistry

Synthesis, Characterization and Application of New Heterocyclic Compounds

A Thesis

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

By

Shahad Mohammed Jassim Al Safy

B.Sc.University Of Karbala 2019

Supervised by

Asst. Prof.Dr.

Nour Abd Alrazzak Abd Allatif

2022 A.D **1444 A.H**

بسم الله الرّحمن الرّحيم

﴿ يَرْفَعِ اللَّهُ الَّذِينَ آمَنُوا مِنْكُمْ وَالَّذِينَ أُوتُوا الْعِلْمَ

دَرَجَاتٍ وَاللَّهُ بِمَا تَعْمَلُونَ خَبِيرٌ ﴾

سورة المجادلة: (11)

Dedication

To lips that always said prayers to me whenever they uttered

My Mother

To my first teacher and model in life

My Father

To Whom they assisted me in life

My sister & brothers

To My Self

Shahad 2022

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 Deanery of College of Science for girls and staff of the department of Chemistry.

My deepest thanks to Prof. Dr.Ayad Fadil, Asst.prof.Dr.Sadiq AbdulHussain, Dr.Ali Talib , Dr. Muna Alturaihi and Assist .Lect. Zainab Hameed for their help in this work .

I would like to thank 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.

Shahad 2022

Supervisor's Certification

I, certify that this research entitled **" Synthesis,characterization and application of new heterocyclic compounds"** was prepared by

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

Signature:

Name: Dr Nour Abd Alrazzak

Scientific Degree: Assistant Professor

Address: College of science for women /Department of Chemistry

Date: 2022 / 7 / 17

Recommendation of Head of Chemistry Department

According to the available recommendation , I forward this thesis for Discussion

Signature of Chemistry Department

Name: Dr.Hazim Yahya Mohammed

Scientific Degree : Assistant Professor

Address : Head of Chemistry Department

University of Babylon / College of Science for Woman

Date: 2022 / 7 / 17

Abstract

This study involves synthesis of new heterocyclic compounds used *p*-aminobenzoicacid and resorcinol as starting materials to produce the intermediates molecules and target molecules. Also this study involves synthesis of new different five and six member heterocyclic compound rings[S4-S11] starting from acid hydrazide [S3] and synthesis of new different Schiff 's base heterocyclic compounds [S24-S29] from 1,3,4-thiadiazole amine [S12]. 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 divded into five different parts :

First part :

It is involve synthesis for azo dye by reaction between *p*-aminobenzoic acid and resorcinol, azo compound [S1] was converted to ester compound [S2] which reacted with hydrazine hydrate to give hydrazide derivative then synthesis of pyridazine-3,6-dione and phthalazin derivatives [S4-S7] from acid hydrazide [S3] as shown in scheme 1.

Second part :

1. It is involve synthesis for pyrazole derivatives [S8-S11] from the reaction of acid hydrazide [S3] with (acetyl acetone, ethyl acetoacetate, methyl acetoacetate and diethylmalonate) in absolute ethanol as shown in scheme 2.

Third part :

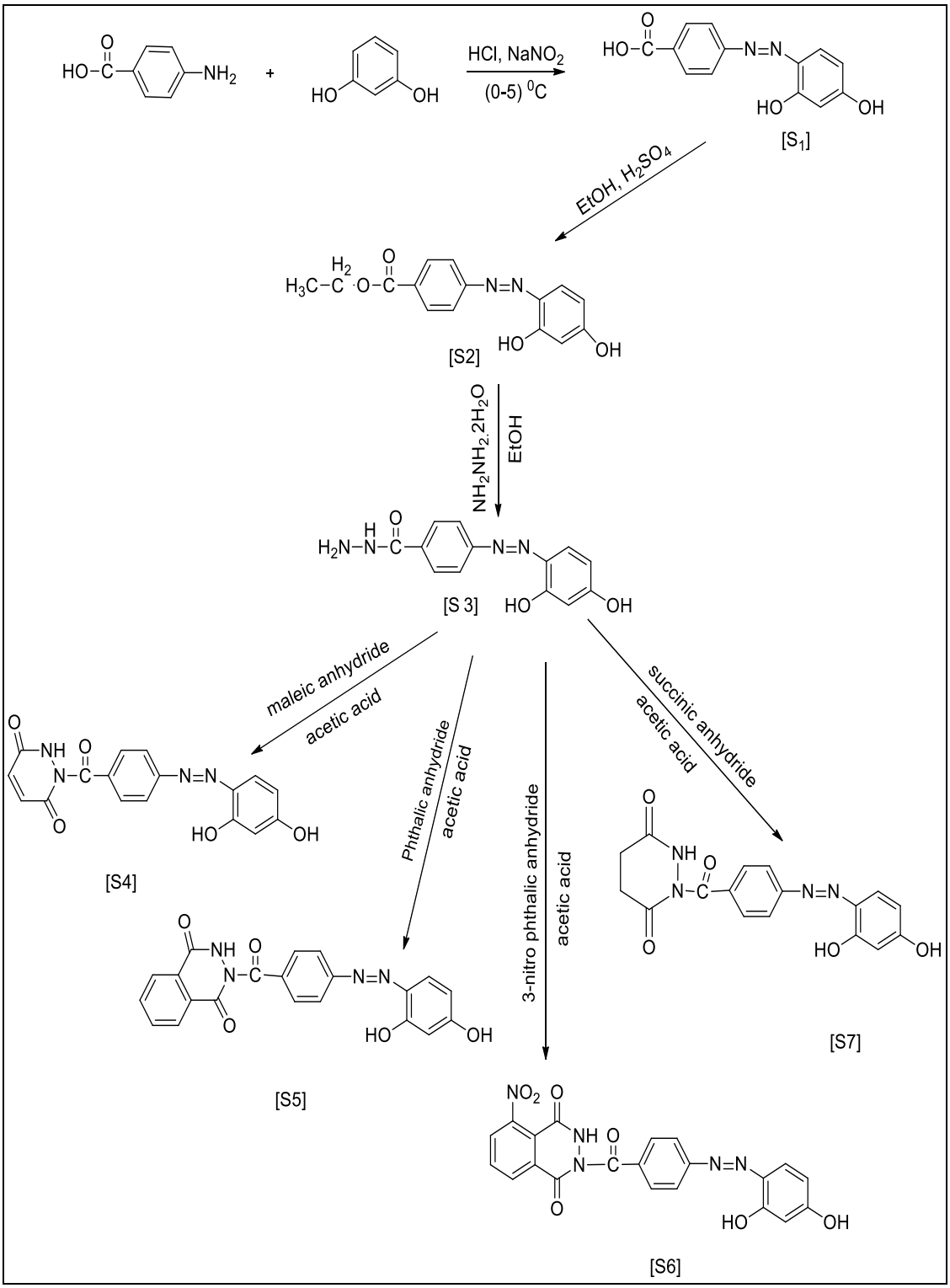
It involves synthesis of 1 ,3 ,4 –thiadiazole amine[S12] by reaction azo dye [S1] with thiosemicarbazide and synthesis new azo dyes [S13-S23] from reaction thiadiazole [S12] with(orcinol ,hydroquinone ,resorcinol, catechol, *α*-naphthol ,*β*-naphthol,2-methyl naphthol,2,6-dimethyl phenol, 1,2,4- triazole ,thiol ,1,2,4 –tri azole and *p*-touldine). as shown in scheme 3.

Fourth part :

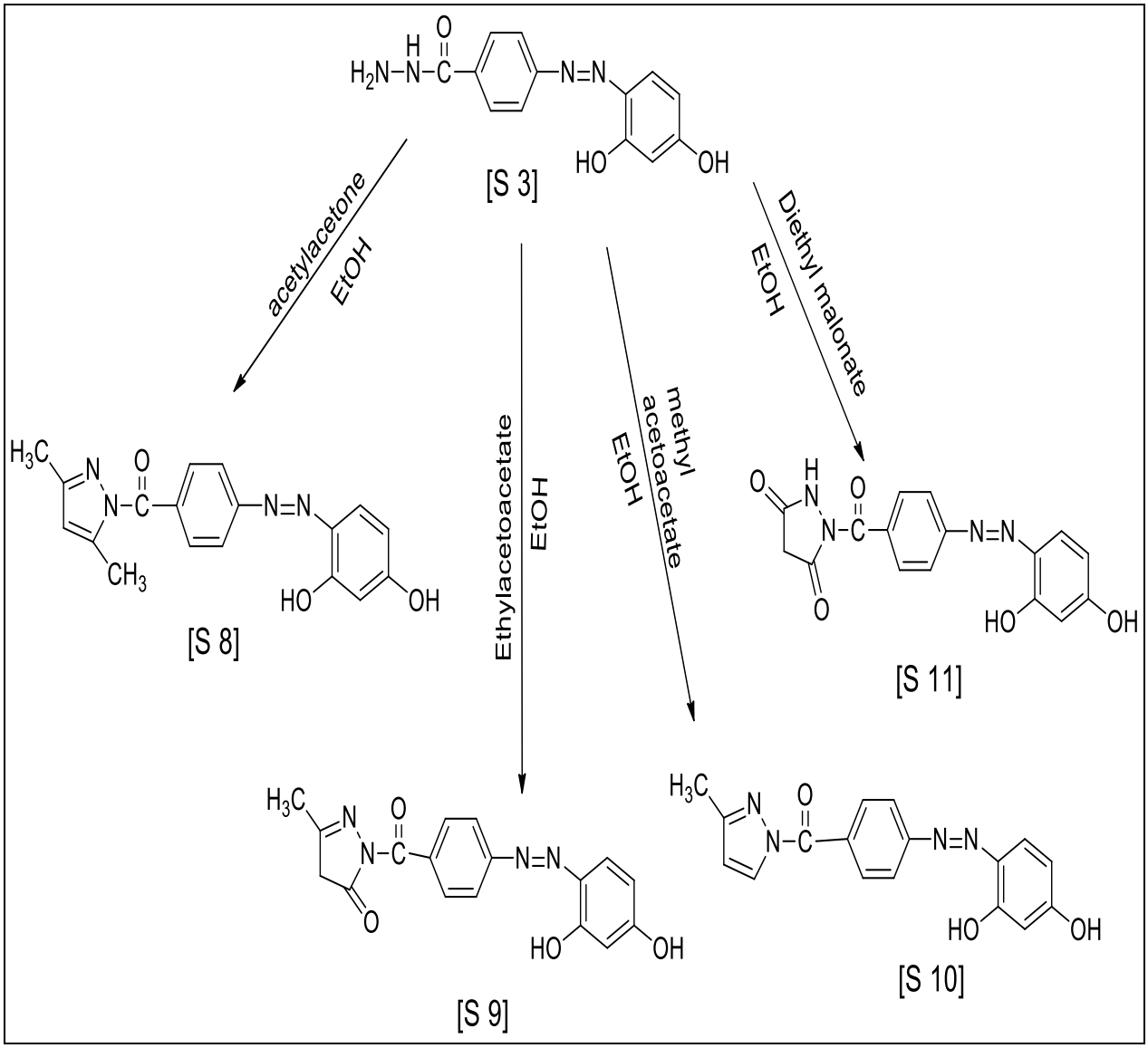
It is involves synthesis of new Schiff 's base compounds [S24-S29] from reaction of 1,3,4- thiadiazole amine [S12] with aromatic aldehydes (9-anthraldehyde, terephthaldehyde ,N,N-dimethyl amino benzaldehyde,3,5-dichlorosalicyaldehyde,3-hydroxybenzaldehdye and 2-hydroxy-5-nitro benzal dehyde) in absolute ethanol as shown in scheme 4.

Fifth part :

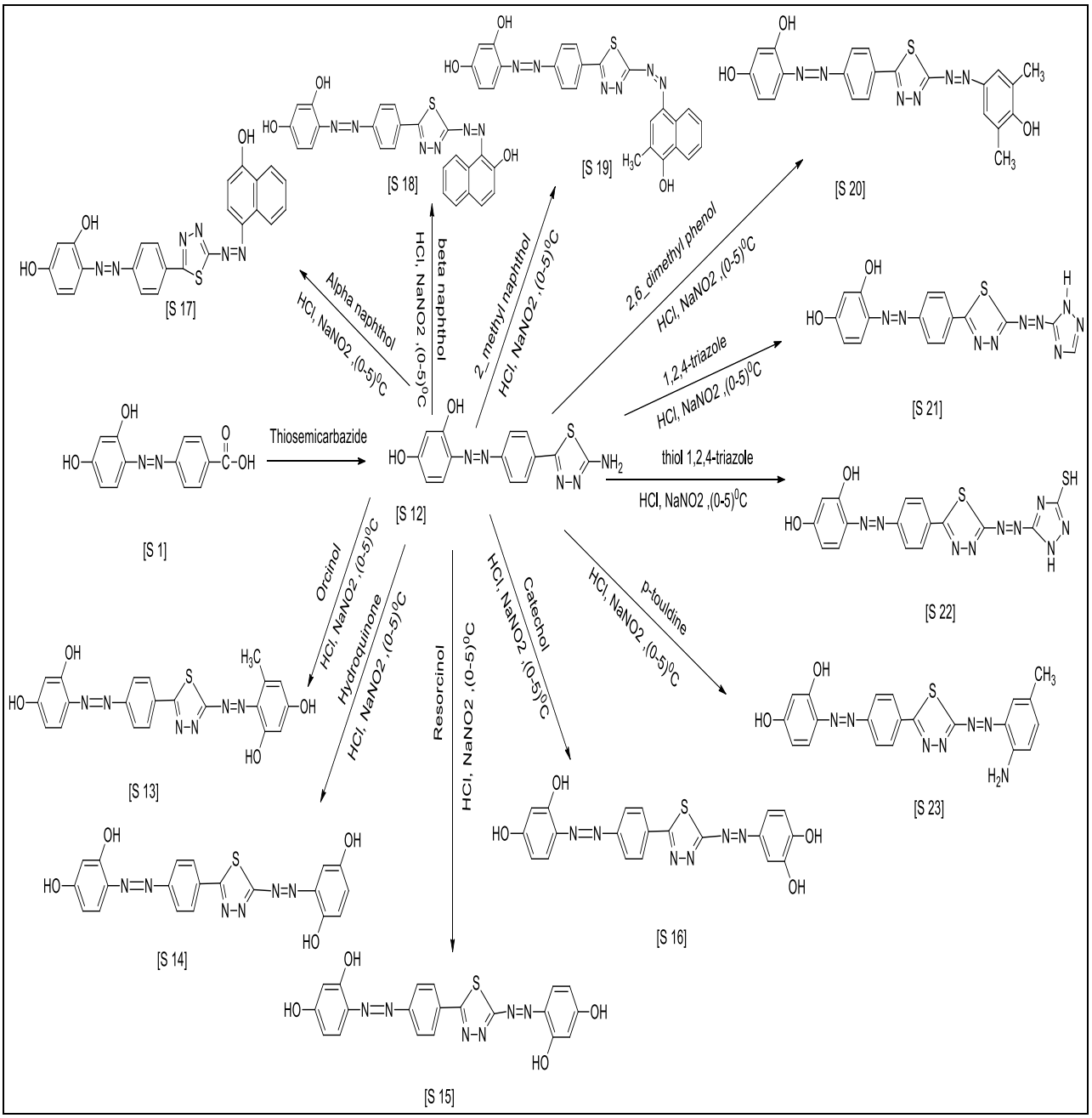
This part involves the study of antibacterial activities of some of the synthesized compounds .These activities were tested by the agar disc- diffusion method against five pathogenic strains of bacteria (*Staphylococcus epiderimidis* and *Staphylococcus aureus* , *Klips* , *pseudomonas* and *E.Coli*) as shown in table 3-1. Also it involves the study of anti-corrosion of some of the synthesized compounds .



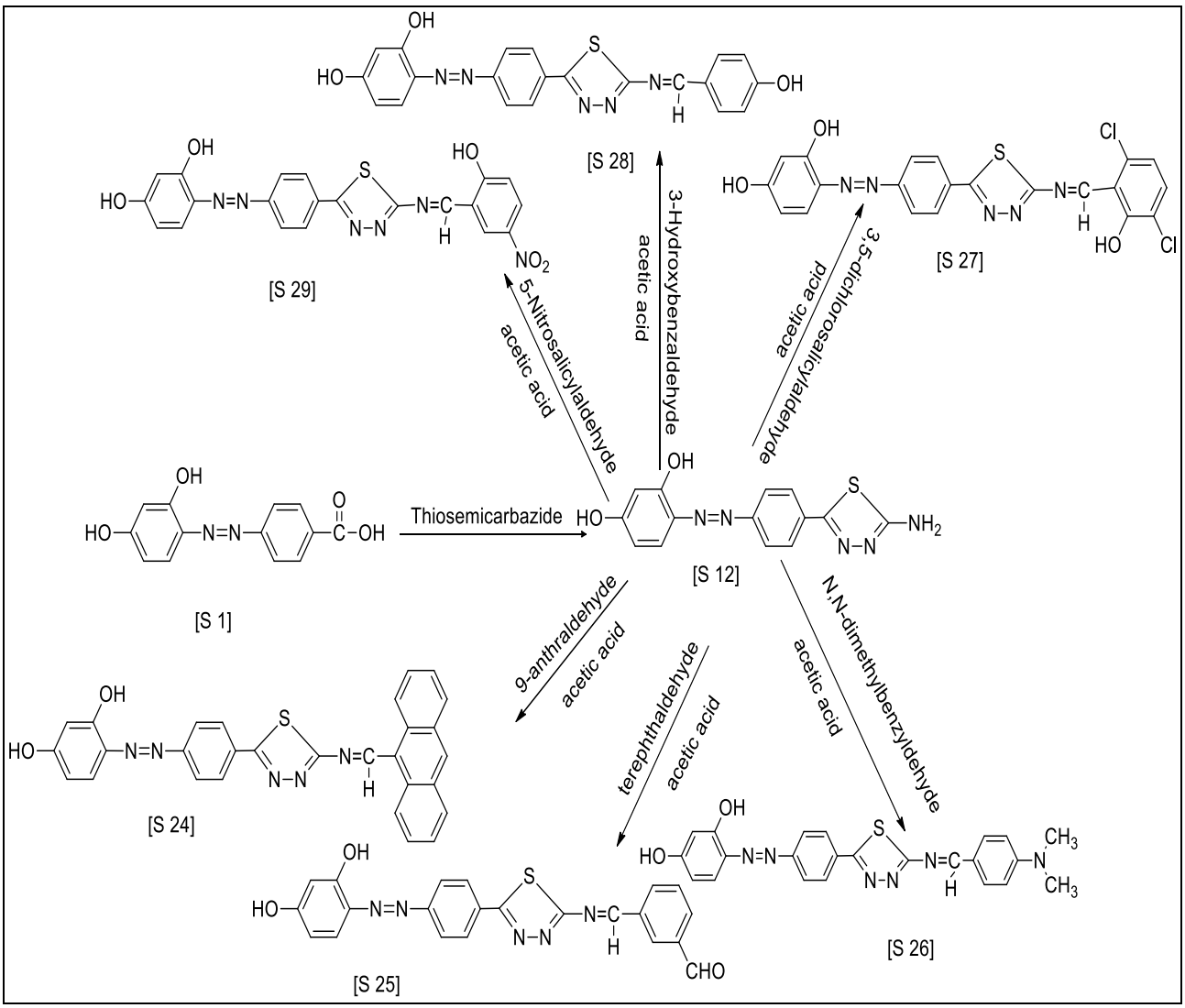
Scheme 1 : Synthesis of Compounds [S1-S7]



Scheme 2 : Synthesis of Compounds [S8-S11]



Scheme 3 : Synthesis of Compounds [S12-S23]



Scheme 4 : Synthesis of Compounds [S24-S29]

List of Contents

Directory of Abbreviations

Symbol	Description
FT-IR	Infrared
^{1}HNMR	Proton nuclear magnetic resonance
^{13}CNMR	Carbon nuclear magnetic resonance
DMSO	Dimethyl sulfoxide
DCM	Dichloromethane
Abs-ETOH	Absolut Ethanol
CHNS	Carbon ,hydrogen ,nitrogen and sulfur
$POCl_3$	Phosphorus oxychloride
TMS	Tetra methyl silane
M.F	Molecular Formula
M.wt	Molecular Weight
M.P	Melting Point
Eocp	establish steady state open circuit potential
IE	The inhibition efficiency
Icorr	corrosion current density
Ecorr	corrosion potential

Chapter one

Introduction

and

Literature Review

1. Introduction

1.1 .Heterocyclic compounds :

Heterocyclic compounds are considered an important branch of organic compounds due to their use in pharmaceuticals, agricultural fertilizers, and industrial investigations[1]. A number of atoms, including N, O, S, P, Si,Se,B and As, can be integrated into the ring structures [2]. Five- or six-membered ring [3].heterocycles are the most prevalent. It has long been established that heterocyclic compounds, which have an element in their ring other than carbon, exhibit vibrant biological activity. Numerous studies have been conducted on the biological effects of heterocyclic rings, including triazoles, indoles, pyrones, morpholines, pyridines, and pyrazoles[4,5].

Because of their biological characteristics, heterocyclic molecules, especially those with sulfur and nitrogen atoms, are a fascinating subject for research in the planning of organic synthesis[6].

thiazole 1,3,4-thiadiazole

Figure (1)

Five-membered nitrogen heterocyclic compounds are thought to be biologically active chemicals and extremely important structural components[7-11].

Additionally, they are crucial in the manufacturing of industrial compounds such as insecticides, corrosion inhibitors, dyes, acid-base indicators, and others [12-16].

Heterocyclic compound membered rings have involves a requirement being placed in the pharmaceuticals and industrial field. 1,3,4- Oxadiazoles are important heterocyclic chemicals used in the production of medicines, polymers, and pigments [17-19].

1.2.Azo Compounds:

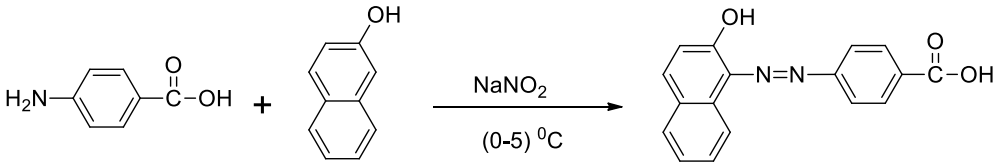
One of the most significant classes of organic chemicals are azo dyes, characterizes by presence (–N=N–) in their structure [20-22].

The structure of azo compounds contains a chromophoric group (azo group -N=N-), and an auxochromic groups like (OH, NH_2, NHR and NR_2) which can be found as substituents on the aromatic rings in the azo compound structure. Because of the bright color of these compounds, azo dyes are used in fabric, plastic industry as paints [23]

Azo dyes have unique biological uses as anti-inflammatory, anti-cancer, anti-diabetic, cleaning agents, and other widely available chemotherapeutic agents. Azo compounds are exceedingly colored and utilized as dyes and Azo hues have outstanding significance in agreement with their environmental stability, electrical, and optical properties. Pigments for a long time [24-26].

1.2.1. Synthesis of Azo Compounds :

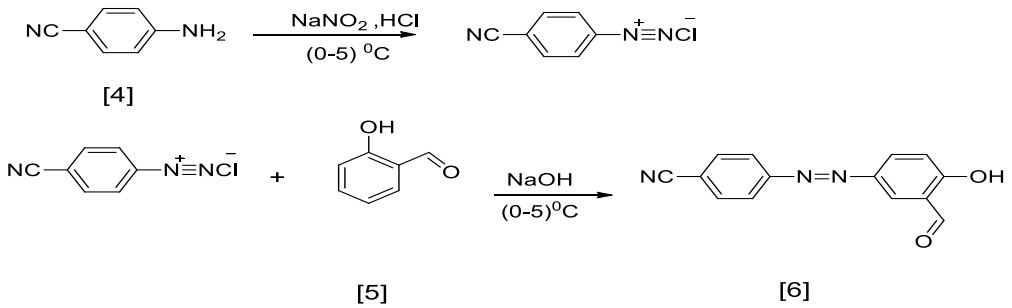
Nour and *et. al*. were prepared compound [3] from reaction *p*-amino benzoic acid [1] with 2-naphthol [2]at (0-5 ^{0}C) [27] .



[1] [2] [3]

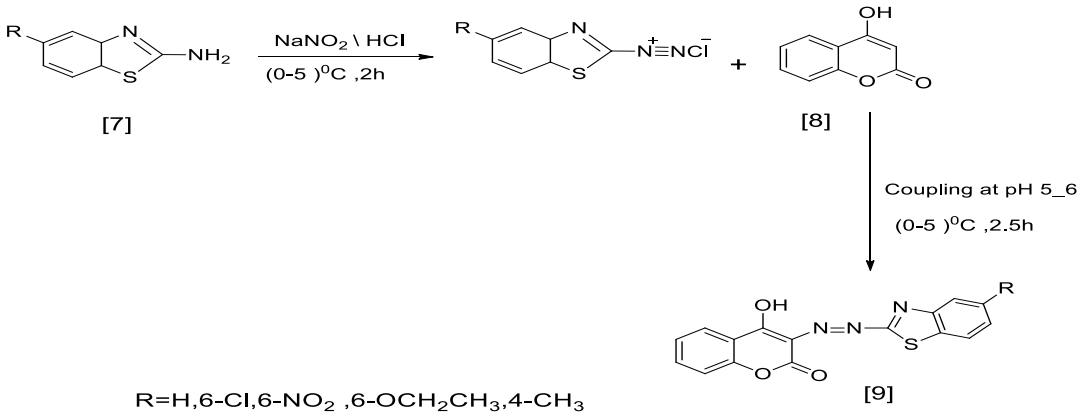
Equation (1)

A.Abdulridha and *et.al*. were prepared the compound [6] from reaction 4-aminobenzonitrile [4] with salicyladehyde [5] at (0-5) ^{0}C[28] .



Equation (2)

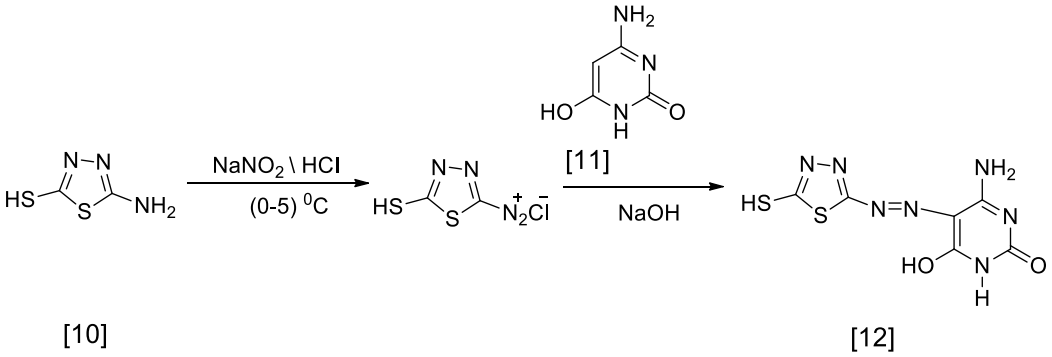
Manjunatha and *et.al*. were prepared compound [9] from reaction 2-aminobenzothiazole [7] with 4-hydroxy coumarin [8] at $(0\text{-}5)^0$C and in presence HCl,$NaNO_2$ then KOH [29].



Scheme (1)

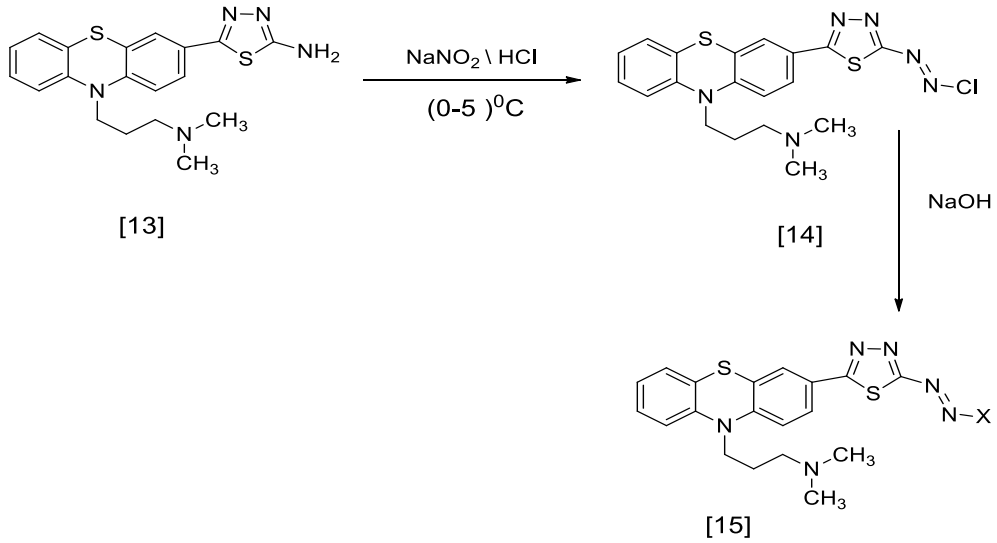
Synthesis of Azo Compounds from Thiadiazole :

J.O.Otutu and *et.al*.were prepared azo compound [12] by reaction 1,3,4-thiadiazole [10] with phenol derivative [11] at $(0\text{-}5)^0$C [30].



Scheme (2)

Chandravadivelu and *et.al*. were prepared azo compound [15] by reaction 1,3,4-thiadiazole[13] with phenol derivative [14] at $(0\text{-}5)^0$C in presence $NaNO_2$,Conc HCl [31].



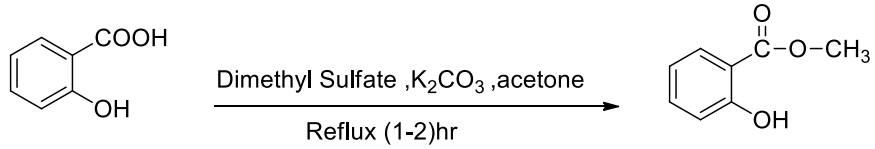
Scheme (3)

1.3.The Esters

Ester is a chemical compound derived from an acid (organic or inorganic) in which at least one –OH hydroxyl group is replaced by an –O–alkyl (alkoxy) group as in the substitution reaction of a carboxylic acid and an al cohol, A carboxylate ester[32].

1.3.1. Synthesis of Ester Compounds :

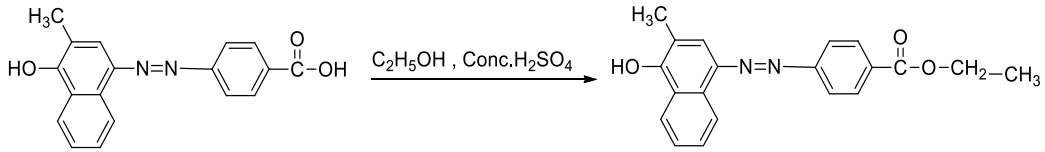
Nageswararao and *et. al*. state that methyl-2- methoxy benzoate [17] was yield from reacted salicylic acid [16] , dimethyl sulfate, potassium carbonate and acetone under reflux (1-2) hour [(33)].



[16] [17]

Equation (3)

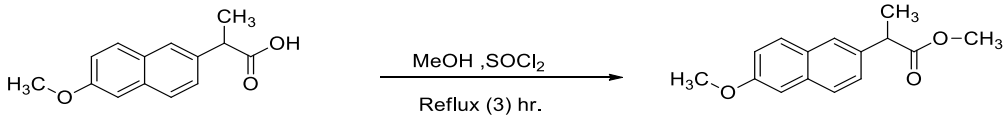
Mohanad and *et.al*. were prepared the compound [19] by reaction the azo compound [18] and absolute ethanol in presence of sulfuric acid and reflux for 4 hours[(34)].



[18] [19]

Equation (4)

M.F.Mahdi and *et.al*.were prepared compound [21] from reaction compound [20] with thionyl chloride then methanol under reflux (3)hours[(35)].



[20] [21]

Equation (5)

1.4.The derivative of Hydrazide:

Hydrazide derivatives are considered biologically active many hydrazide compounds were used in treatment of tuberculosis[(36)],this sort of compound's structural formula is (RCONHNH-) some of carboxylic acid hydrazides were reported to have antimicrobial activities as compound below.

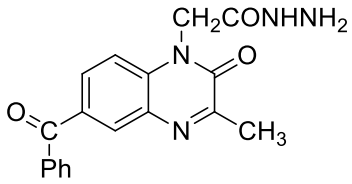
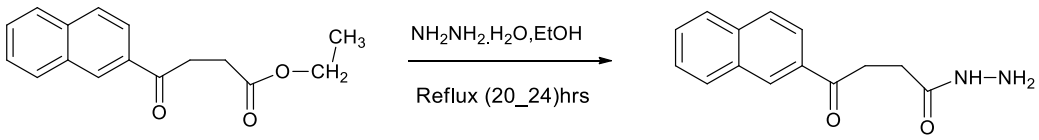


Figure (2)

1.4.1 Synthesis of hydrazide derivatives compounds

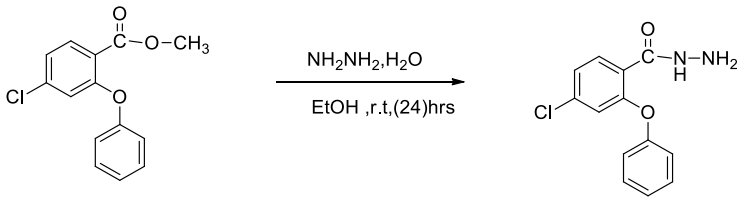
4-Naphthalen-2-yl-4-oxo-butyric acid hydrazide[23]was created, according to Kumar et al. by reacting 4-Naphthalen-2-yl-4-oxo-butyric acid ethyl ester[22] with hydrazine in the presence of ethanol as a solvent under reflux for (20-24) hours[(37)].



[22] [23]

Equation (6)

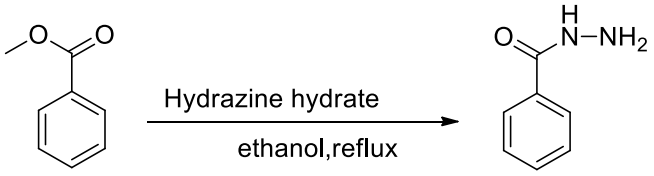
M.Faizi and *et.al*.were prepared the compound[25] from reaction of ester compound [24] with hydrazine hydrate and reflux for overnight at room temperature [(38)].



[24] [25]

Equation (7)

Ahmed A.Saheeb and Wasan K.Damdoom synthesized the compound benzohydrazide [27] by reaction methyl benzoate [26] with hydrazine hydrate and ethanol with reflux for (21-24) hours[(39)] .



[26] [27]

Equation (8)

1.5.0 Pyridazine :

The chemical name for pyridazine is $(CH)_4N_2$. It is a heterocyclic organic molecule. It is aromatic and has a six-membered ring with two nitrogen atoms that are close to one another. It is isomeric with the $(CH)_4N_2$ rings of pyrimidine and pyrazine, respectively [6].

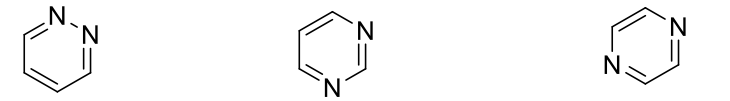


Figure (3)

Various synthetic pyridazine derivatives have been used in agrochemical ,pharmaceutical and other applications [40].

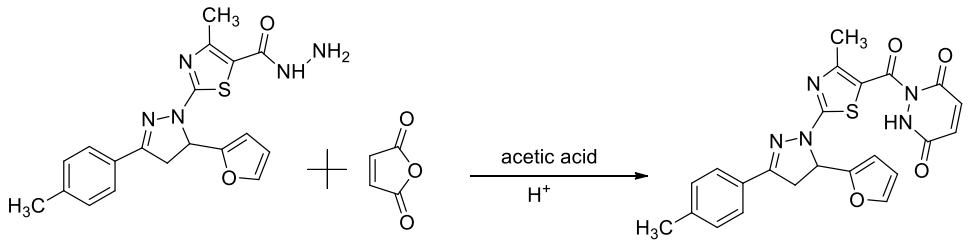
Pyridazine ring can be fused with a benzene ring in two ways giving

phthalazine or cinnoline[41].

Figure (4)

1.5.1. Synthesis of Pyridazine and it's Derivatives:

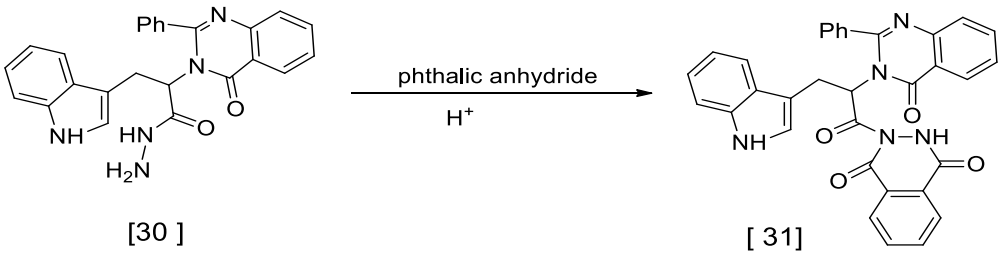
Abdo O.abdelhamid and *et .al*.were prepared the compound [29] from reaction acid hydrazide [28] with maleic anhydride in presence glacial acetic acid and reflux for 2 hours[42].



[28] [29]

Equation (9)

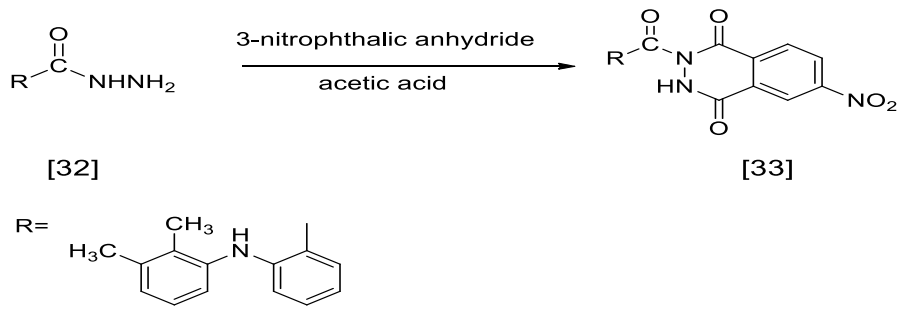
A.H.Samir was prepared the compound [31] by reaction the acid hydrazide [30] with phthalic anhydride in presence glacial acetic acid reflux for 6 hours[43] .



[30] [31]

Equation (10)

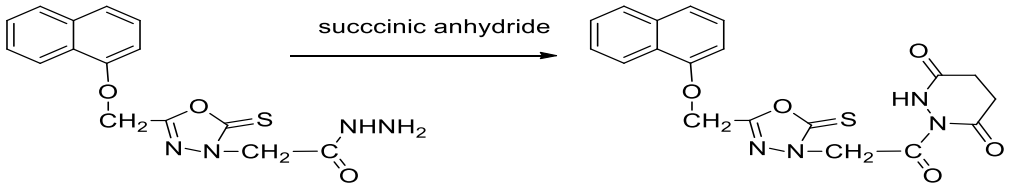
A.H.Ahmed and I. k.Jassim were prepared compound [33]from reaction acid hydrazide [32] with 3-nitrophthalic anhydride in acetic acid and reflux for 8 hours [6].



[32] [33]

Equation (11)

Abdelmotaal and *et. al*. were prepared the compound [35] by reaction acid hydrazide [34] with suucinic anhydride in presence n- butanol and reflux for 6 hours[44] .

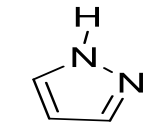


[34] [35]

Equation (12)

1.6.0 Pyrazole:

Three carbon atoms and two nitrogen atoms make up the five membered ring structure of the unsaturated heterocyclic organic molecule pyrazole[6].



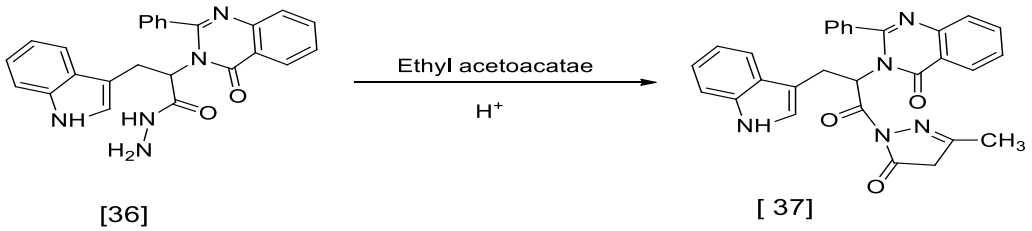
pyrazole

Figure (5)

The pyrazoles class of heterocyclic compounds, there recognized for their significance as drug targets and therapeutic interest. Organic compounds with additional S and or N atoms exhibit a wide range of biological activities, including antibacterial and antioxidant properties [45-49]. Pyrazole core is extremely important in the pharmaceutical sector for the design of drugs such as anticancer, antibacterial, and antifungal because of its medical importance [50-53].

1.6.1 Synthesis of Pyrazole:

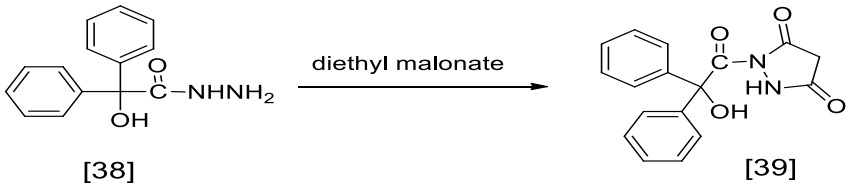
A.hammdi Was prepared the compound [37] by reaction the acid hydrazide [36] with ethylacetoacetate in presence glacial acetic acid and reflux for 6 hours[43] .



[36] [37]

Equation (13)

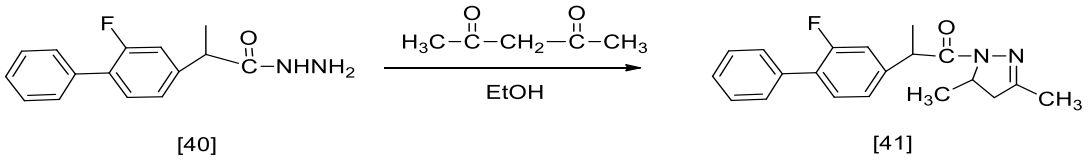
Salim and *et.al*.were prepared the pyrazole [39] by reaction the acid hydrazide [38] with diethyl malonate in absolute ethanol and reflux for 2 hours [54] .



[38] [39]

Equation (14)

M.Amir and Sh.Kumar were prepared the compound [41] by reaction the acid hydrazide [40] with acetyl aceton in absolute ethanol and reflux for 8 hours[55] .



[40] [41]

Equation (15)

1.7. 1,3,4-Thiadiazole:

Thiadiazoles contain a ring-shaped unsaturated structure with the moiety formula $C_2H_3N_3S$ and two carbon, three nitrogen, and one sulfur atoms [56].

Thiadiazoles are uncommon five-membered heterocyclic structures in nature. In such a ring, nitrogen, sulfur, and carbon atoms can be positioned in a variety of ways, giving birth to a number of isomers, including 1,2,3, 1,2,4, 1,2,5, and 1,3,4-thiadiazole [57].

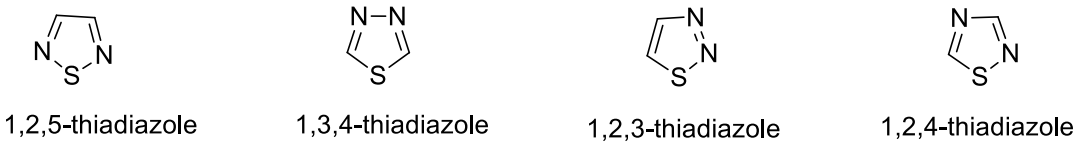


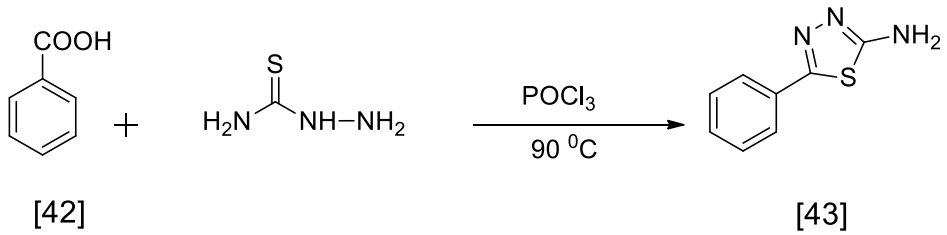
Figure (6)

Literature supports indicates that thiadiazole and their derivatives have been board range biological actions including antifungal ,antimicrobial, anti inflammatory ,antitubercular, anti-HIV , anticonvulsant , anticancer antidiabetics and antiviral activities .Many drugs containing 1, 3, 4- thiadiazole moiety are commercial available in medicine stores such as acetazolamide [58-63].

1.7.1 Synthesis of 1,3,4-Thiadiazole :

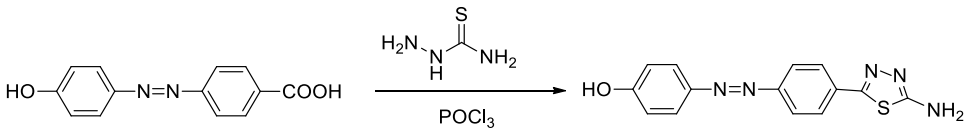
Chinnagiri and *et.al* were prepared 1,3,4-thiadiazole[43] by the reaction benzoic acid [42] with thiosemicarbazide presence of $POCl_3$ reflux for 8 hours [64].

Equation (16)



[42] [43]

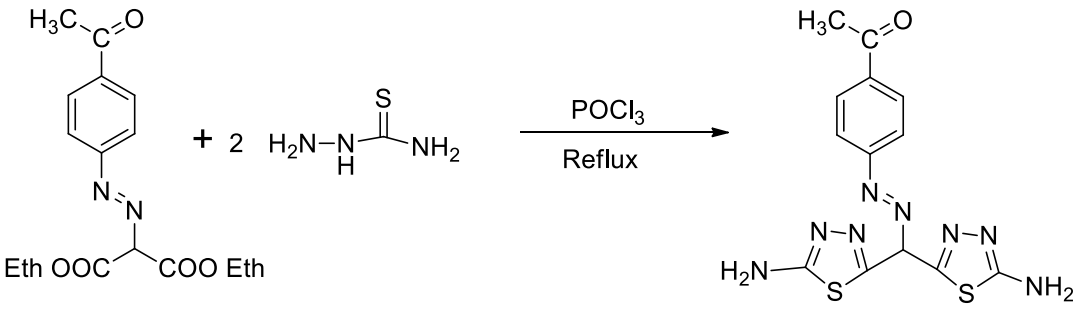
Shimaa and *et.al*. were prepared 1,3,4- thiadiazole [45] by reaction azo compound [44] with thiosemicarbazide in presence of $POCl_3$ and reflux for 4 hours [65].



[44] [45]

Equation (17)

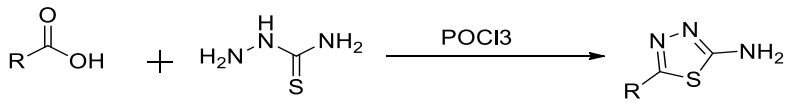
Nagham and *et.al*. were prepared 1,3,4-thiadiazole [47] by reaction azo compound [46] with thiosemicarbazide in presence of $POCl_3$ and reflux for 7 hours [66].



[46] [47]

Equation (18)

Tatiana and *et.al*.were prepared 1,3,4-thaidiazole [49] by reaction carboxylic acid [48] with thiosemicarbazide in presence of $POCl_3$ and reflux for 10 hours [67].



[48] [49]

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

Equation (19)

1.8.Schiff Bases :

Schiff bases are currently being studied extensively due to their ease of synthesis, various structures with wide range of color, and they have sparked a lot of interest in the thermochromic material field [68].

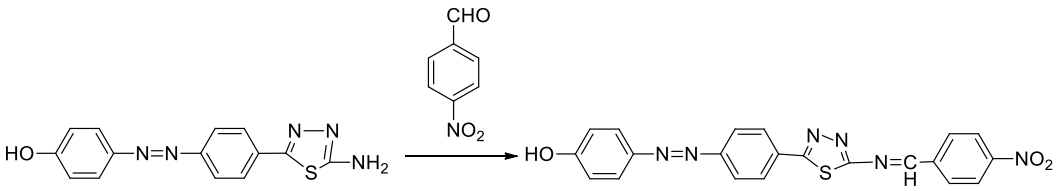
Schiff bases are condensation products of primary amines and carbonyl compounds where imine or azomethine (C=N) groups are used in place of the carbonyl group. They are generally represented by formula $R_1HC=NR_2$ where R_1 and R_2 are alkyl or aryl groups[69-72].

Schiff bases have become more significant in the medical and pharmaceutical areas in many different domains, including analytical, biological, and inorganic

chemistry, Schiff bases are among the most frequently utilized classes of organic molecules. Due to their wide range of biological like activities and anti-inflammatory [73].

1.8.1 Synthesis of Schiff Base Compounds :

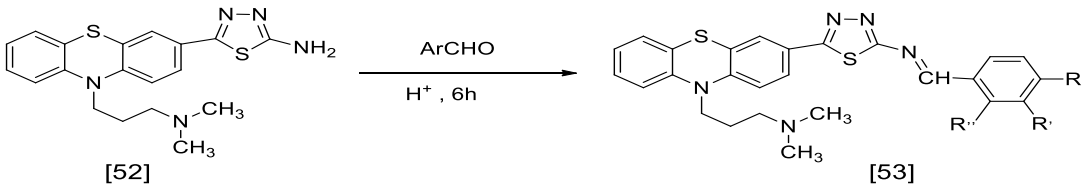
Shimaa and *et.al*. were prepared Schiff base [51] by reaction 1,3,4-thiadiazole[50] with aldehyde derivative and reflux for (2-3) hours [65].



[53] [54]

Equation (20)

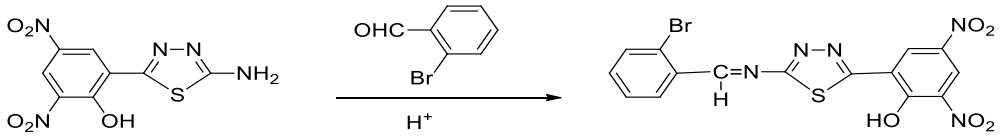
Chandravadivelu and *et.al*.were prepared shciff base [53] by reaction 1,3,4-thiadiazole [52] with aromatic aldehyde and glacial acetic acid under reflux for 6 hours[31].



[52] [53]

Equation (21)

R.kais and S.adnan were prepared shciff base [55] by reaction 1,3,4-thiadiazole [54] with aromatic aldehyde in presence of glacial acetic acid and reflux for 6 hours[74].



[54] [55]

Equation (22)

1.9 The Biological Activity :

Azo dyes have unique biological uses as anti-inflammatory, anti-cancer, anti-diabetic, cleaning agents, and other widely available chemotherapeutic agents. Azo compounds are exceedingly colored and utilized as dyes Pigment for a very long time, but Azo baes have exceptional significance in agreement with their environmental stability, electrical, and optical qualities. [75-77].

Azo dye compounds have a wide range of industrial applications, including photodynamic treatment, are photosensitive, and have biological activity as a result of their use in inflammatory ,anticancer and antibacterial processes and antifungal [78].

The activity of pyridazine derivatives in the field of analgesics and inflammation, antibiotics, epilepsy, cardiovascular diseases, and oncology has been reviewed recently[79-83].

Pyrazole core is extremely important in the pharmaceutical sector for the development of drugs that are anticancer, antibacterial, antimicrobial, antifungal, etc. because of its medicinal usefulness [84-86].

One of the most popular heterocyclic pharmacophores is 1,3,4-thiadiazole. They exhibit a wide range of biological actions, including acetyl cholinesterase inhibition for the treatment of Alzheimer's disease, antibacterial, anticancer, antioxidant, antidepressant, anticonvulsant, and antihypertensive effects [87].

Schiff bases have been shown to provide a variety of biological functions. Antifungal, antibacterial, antimalarial, anti-proliferative, anti-inflammatory, antiviral, and antipyretic characteristics are among these biological actions [64].

1.10 Corrosion and Its Effect :

The term "corrosion" refers to the damage of metals and alloys caused by chemical or electrochemical contact with their environment[88]. Wet and dry corrosion are the two categories into which corrosion reactions are categorized in terms of the types of corrosive environments[89,90].

Every use of metals is affected by the problem of corrosion. Corrosion damage raises the expense of maintaining and protecting the materials used. The difficulty for scientists working in this field is the development of techniques to control corrosion. Use of an inhibitor is one of the appealing and most useful corrosion control techniques available for metals coming into contact with corrosive media. By limiting the dissolving and consumption of metal, inhibitors lessen the corrosion of metallic materials. Organic compounds with nitrogen, sulphur, oxygen, or N - heterocyclic compounds with polar groups make up the majority of the well-known inhibitors for the corrosion of steel in acidic environments. Corrosion inhibitors for mild steel in acidic conditions have been the topic of numerous research studies [91].

Although there are several ways to prevent or reduce corrosion, using inhibitors is typically used to safeguard metals from doing so, especially in closed systems.

The existence of distinctive structural elements such as I the type of functional groups, (i) aromatic rings, (ii) donor heteroatoms like N, O, and S, (iii) molecular planarity, (iv) electron distribution, and (v) steric variables is necessary for a corrosion inhibitor to operate well.The usage of several organic compounds bearing heteroatoms as efficient inhibitors for the corrosion control of mild steel in an acidic environment is suggested by literatures [92,93].

Aim of Work :

1-Synthesis and characterization of new heterocyclic compounds derivatives and this involve :

a-Synthesis of new azo compounds and ester.

b-Synthesis of new hydrazide derivatives.

c-Synthesis of new pyrazole , pyradazine and phthalazine.

d- Schiff base and 2-amino 1,3,4-thiadiazole were synthesized.

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

3- Study the corrosion for 2-amino 1,3,4-thiadiazole and some new azo dyes .

Chapter Two

Materials and Methods

2.Experimental and Methods

2.1. Chemicals:

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

Chemicals	Supplier	Purity%
Acetic acid	CDH	99
Acetyl acetone	Sigma-Aldrich	99.3
Alpha naphthol	Sigma-Aldrich	98
p-amino benzoic acid	CDH	99
9-Anthraldehyde	Merk	99
Catechol	CDH	99
3,5-dichlorosalicyladehyde	Sigma-Aldrich	98
2,6-dimethyl phenol	Merk	99
Diethylmalonate	Merk	98
DMSO	BDH	99
Ethanol	CDH	99.8
Ethyl acetoacetate	BDH	99
Hydrazine hydrate	CDH	80
Hydrochloric acid	CDH	37
Hydroquinone	Merk	99
3-hydroxy benzaldehyde	Sigma-Aldrich	99
2-hydroxy-5-nitrobenzaldehyde	Merk	98
Maleic anhydride	CDH	99
Methyl acetoacetate	Merk	98
2-methyl naphthol	Merk	99
Beta naphthol	Sigma-Aldrich	98
3-nitrophthalic anhydride	Merk	99
N,N-dimethyl aminobenzaldehyde	Merk	98
Orcinol	CDH	98

Phthalic anhydride	CDH	99
Phosphoryl chloride	CDH	99
Potassium hydroxide	CDH	98
Resorcinol	CDH	98
Sodium hydroxide	CDH	97
Sodium nitrite	Merk	99
Succinic anhydride	CDH	99
Sulfuric acid	Merk	99
Terephthaldehyde	Sigma-Aldrich	98
Thiosemicarbazide	CDH	99
P-Toluidine	Merk	99
1,2,4-Triazole	Merk	98
1,2,4-Triazole-3-thiol	Merk	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 .

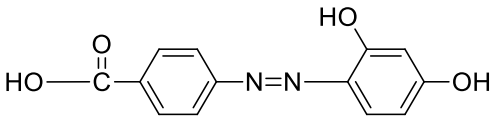
3- ^{1}H-NMR Spectra and ^{13}C-NMR Spectra was verified on a Bruker (AC 400) NMR spectrometer, working at(500 MHz) for ^{1}H-NMR and (126MHz) for ^{13}C-NMR ,Iran. All chemical shifts (δ) are reported in ppm relative to tetramethylsilane (TMS) as references (δ=0.0 ppm),CHNS was used for characterization of the prepared compounds .

4-The biological activity was performed by Microbiology Department , college of science \ Babylon University

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

2.3. Methods :

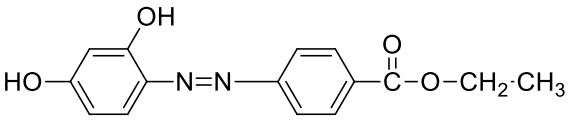
2.3.1. Synthesis of Compound [S1]:



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,1.10g) resorcinol in distill water 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 [94].

Compound [S1]: molecular formula:$C_{13}H_{10}N_2O_4$, color :orange,yield :93 % , m .p = 195-197 ^{0}C.

2.3.2. Synthesis of Compound[S2]:

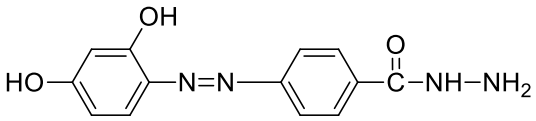


(0.01mole, 2.58g) of compound [S1] dissolved in absolute ethanol and (8 ml) Conc.H_2SO_4 was added as drop wise to the solution The mixture was refluxed

for 24 hours , then the reaction content is poured into ice water ,filtered and washed with water the precipitate was recrystallized by absolute ethanol [94].

Compound[S2]: molecular formula: $C_{15}H_{14}O_4N_2$, color: orange, yield: 93%, m. p = 184-186 ^{0}C.

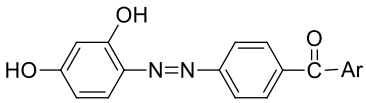
2.3.3. Synthesis of Compound [S3]:



To ethanolic solution of (0.01 mole, 2.86g) of ester, added(0.01 mole, 0.48 ml) 80% hydrazine hydrate a drop wise. The solution was refluxed for 24 hours, after that poured in ice water and the solvent was evaporated , the precipitate was recrystallized by absolute ethanol [94].

Compound[S3] molecular formula:$C_{13}H_{12}N_4O_3$, color: black , yield: 85 %, m .p = 75-78 ^{0}C

2.3.4. Synthesis of Compounds [S4-S7]:



Ar =(maleic anhydride , phthalic anhydride ,3-nitrophthalic anhydride ,succinic anhydride.)

Compound [S3] (0.01mole, 2.72g) was mixed with (0.01mole, 0.98g, 1.48g, 1.93g, 1g) of compounds (maleic anhydride, phthalic anhydride, 3-nitrophthalic anhydride, succinic anhydride) respectively in glacial acetic acid (0.57 ml).the mixture was refluxed for 7 hours then cooled and added onto crushed ice , The precipitate were filtered out and the final products were washed with water, and recrystallized by absolute ethanol [41].

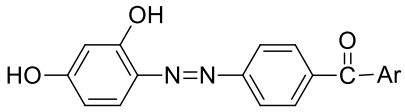
Compound [S4] molecular formula : $C_{17}H_{12}N_4O_5$, color: light brown ,yield : 78% , m .p =112-115 ^{0}C

Compound [S5] molecular formula:$C_{21}H_{14}N_4O_5$, color: dark brown ,yield: 88 %, m .p=91-93^{0}C

Compound [S6] molecular formula :$C_{21}H_{15}N_5O_7$,color: light brown ,yield: 81 %, m .p =120-122 ^{0}C

Compound [S7] molecular formula : $C_{17}H_{14}N_4O_5$,color: brown ,yield: 77 %, m .p =138-140 ^{0}C

2.3.5.Synthesis of Compounds [S8-S11]:



Ar = (acetyl acetone, ethyl acetoacetate, methyl acetoacetate ,diethylmalonate)

Mixture of hydrazide [S3] (0.01 mole, 2.72 g), (0.01 mole, 1.025 ml, 1.26 ml, 1.078 ml, 1.51 ml) of compounds (acetyl acetone, ethyl acetoacetate, methyl acetoacetate ,diethylmalonate) respectively in absolute ethanol were heated, under reflux temperture for 13 hours. The mixture of reaction were cooled and the formed precipitate were filtered off for giving the final the precipitate was recrystallized by absolute ethanol[41].

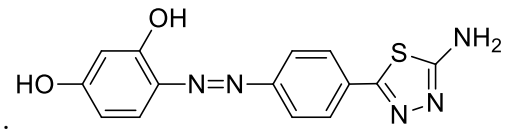
Compound [S8] molecular formula: $C_{17}H_{14}N_4O_4$,color: light brown ,yield: 82 %, m .p = 120-122 ^{0}C

Compound [S9] molecular formula: $C_{18}H_{16}N_4O_3$, color: dark brown ,yield: 80 %, m .p =107-109 ^{0}C

Compound [S10] molecular formula: $C_{17}H_{14}N_4O_3$,color: brown ,yield: 81 %, m .p =95-99 ^{0}C

Compound [S11] molecular formula : $C_{19}H_{11}N_5O_2S_1$,color: dark brown ,yield: 76 %, m .p =111-113 ^{0}C

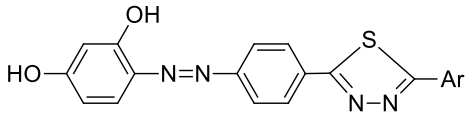
2.3.6.Synthesis of compound [S12]:



(0.01mole, 2.58g) of compound [S1] 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 [94]

compound [S12]: molecular formula: $C_{14}H_{11}N_5O_2S_1$, color: brown , yield: 78%, m .p=180-182^{0}C

2.3.7.Synthesis of compounds [S13-S23]:



(Ar= orcinol ,hydroquinone ,resorcinol, catechol, alpha naphthol ,beta naphthol,2-methyl naphthol,2,6-dimethyl phenol,1,2,4-triazole,thiol 1,2,4-triazole.p-touldine)

(0.003mole, 1g) Thiadiazole derivative was dissolved in (13ml) distilled water and (2.5mL) HCl , and the temp. at (0-5 °C) . Then (0.003 mole, 0.20g) of $NaNO_2$ which dissolved in (6 ml) distilled water were added as drop wise to the solution for (15) min.Diazonium solution were added as drop wise to coupling component solution which prepared by mixing (0.003 mole, 0.37g ,0.33g,0.33g ,0.33g ,0.43g ,0.43g, 0.47g ,0.36g ,0.20g, 0.30g , 0.32g) of compounds (orcinol ,hydroquinone ,resorcinol, catechol, α naphthol ,β naphthol,2-methyl naphthol,2,6-dimethyl phenol, 1,2,4-triazole,thiol 1,2,4-triazole,*p*-touldine) respectively in distill water and (0.8 g)

of sodium hydroxid in(8ml) distilled water. The precipitate then filtered and washed with water and was recrystallized by absolute ethanol [94].

Compound [S13] molecular formula :$C_{21}H_{16}N_6O_4S$,color: dark brown ,yield:83 , %, m .p =92-95^{0}C

Compound [S14] molecular formula : $C_{20}H_{14}N_6O_4S$,color: black , yield: 85%, m .p =90-93^{0}C

Compound [S15]molecular formula : $C_{20}H_{14}N_6O_4S$,color: black , yield: 86%, m .p =165-168^{0}C

Compound [S16] molecular formula : $C_{20}H_{14}N_6O_4S$,color: dark brown , yield:87 %, m .p =180-182^{0}C

Compound [S17] molecular formula : $C_{24}H_{16}N_6O_3S$,color: light brown , yield: 83%, m .p =190-192^{0}C

Compound [S18] molecular formula : $C_{24}H_{16}N_6O_3S$,color: dark brown , yield: 84%, m .p =117-120^{0}C

Compound [S19] molecular formula : $C_{25}H_{18}N_6O_3S$,color: brown , yield: 87%, m .p =95-98^{0}C

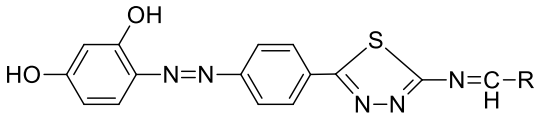
Compound [S20] molecular formula : $C_{22}H_{18}N_6O_3S$,color: light brown ,yield: 86%, m .p =110-113^{0}C

Compound [S21] molecular formula : $C_{16}H_{11}N_9O_2S$,color : dark brown , yield: 85%, m .p =92-94 ^{0}C

Compound [S22] molecular formula :$C_{16}H_{11}N_9O_2S_2$,color: brown ,yield: 88%, m .p = 91-93^{0}C

Compound [S23] molecular formula : $C_{21}H_{17}N_7O_2S$,color: light brown , yield:77 %, m .p =88-90 ^{0}C

2.3.8.Synthesis of compounds [S24-S29]:



(R=9-anthraldehyde,terephthaldehyde,N,N-dimethyl amino benzaldehdye,3,5-dichloro salicyaldehyde,3-hydroxy benzaldehdye,2-hydroxy-5-nitrobenzaldehyde)

(0.003mole, 1g) Of compound [S12] was dissolved in (25ml) absolute ethanol, and two drops of glacial acetic acid were added, followed by addition (0.003 mole, 0.618g, 0.402g, 0.44g, 0.573g, 0.36g, 0.50g) of compounds (9-anthraldehyde, terephthaldehyde ,N,N-dimethyl amino benzaldehyde,3,5- , dichlorosalicyaldehyde,3- hydroxybenzaldehdye,2-hydroxy-5-nitrobenzal dehyde) respectively the combination was refluxed for 14 hours, after which the solvent was evaporated and the result was collected and recrystallized from absolute ethanol[94].

Compound [S24] molecular formula : $C_{29}H_{19}N_5O_2S$,color: light brown ,yield: 87 %, m .p =192-194 ^{0}C

Compound [S25]molecular formula :$C_{22}H_{15}N_5O_3S$,color: light brown ,yield: 82 %, m .p =188-190 ^{0}C

Compound [S26] molecular formula : $C_{23}H_{20}N_6O_2S$, color: dark brown , yield: 75%, m .p =180-182 ^{0}C

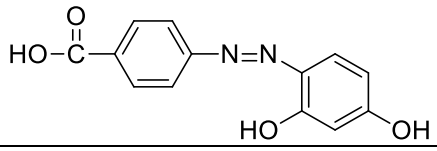
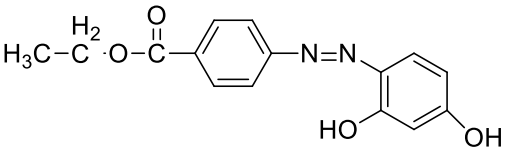
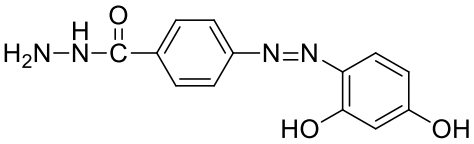
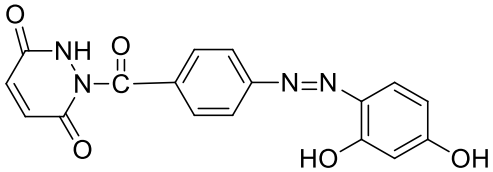
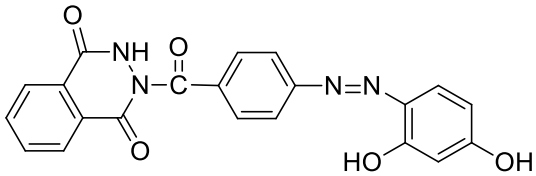
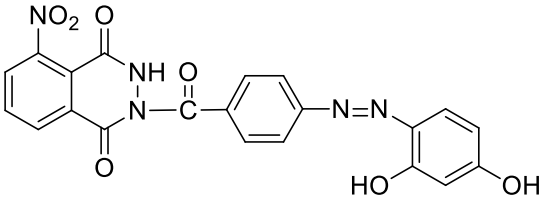
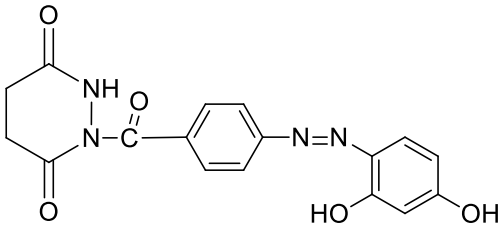
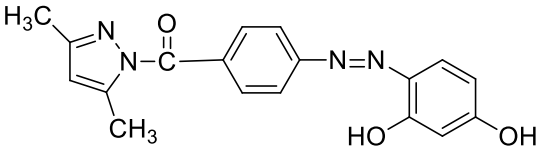
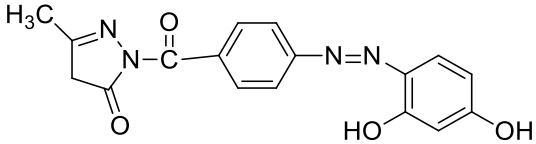
Compound [S27] molecular formula :$C_{21}H_{13}Cl_2N_5O_3S$, color: brown ,yield: 86 %, m .p =183-186 ^{0}C

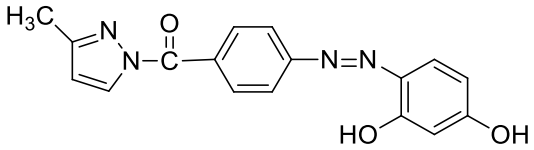
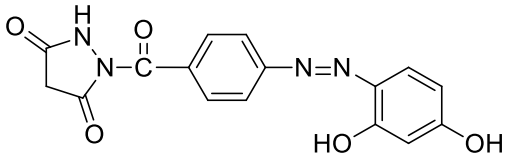
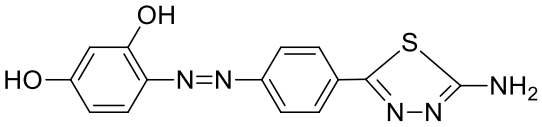
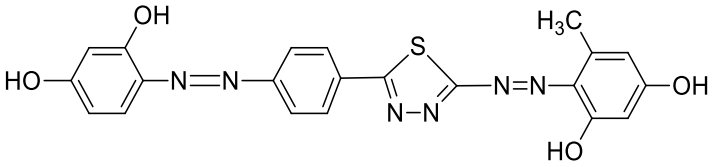
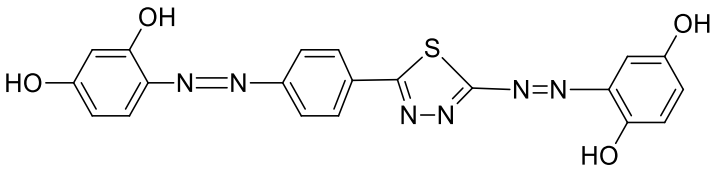
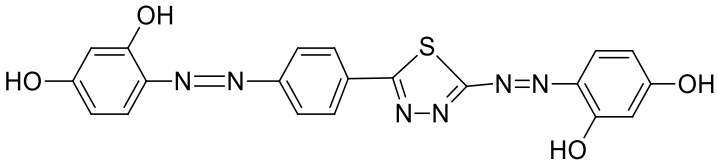
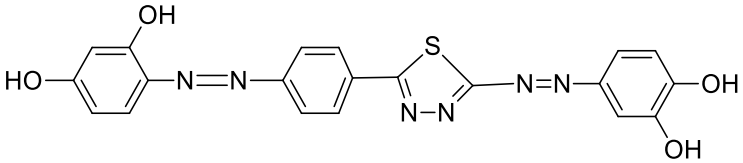
Compound [S28] molecular formula : $C_{21}H_{15}N_5O_3S$, color: black ,yield: 80%, m .p =190-192 ^{0}C

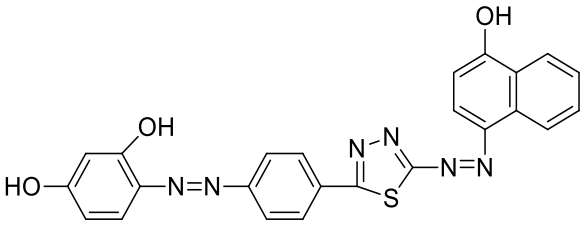
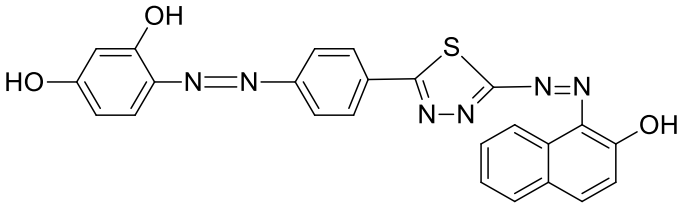
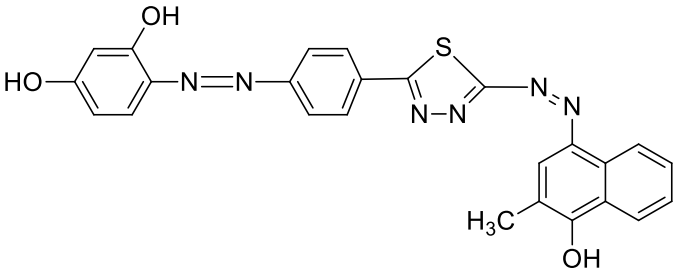
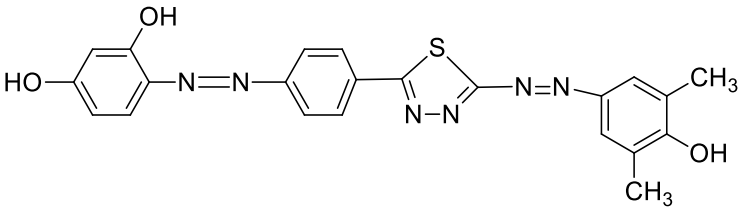
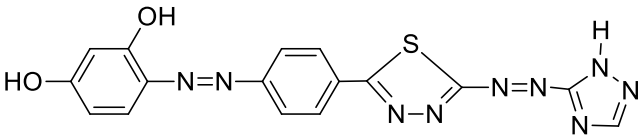
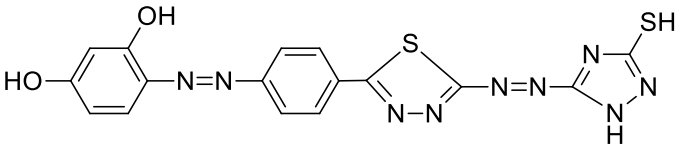
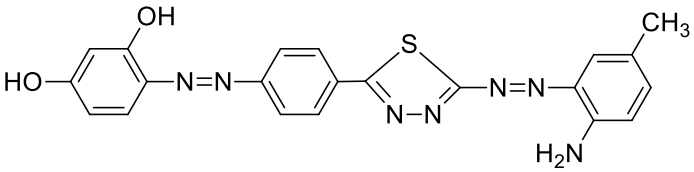
Compound [S29]molecular formula : $C_{21}H_{14}N_6O_5S$,color: light brown , yield: 74 %, m .p = 192-194 ^{0}C

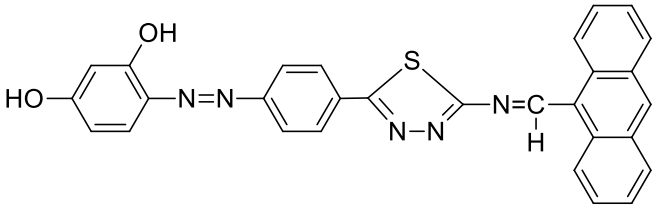
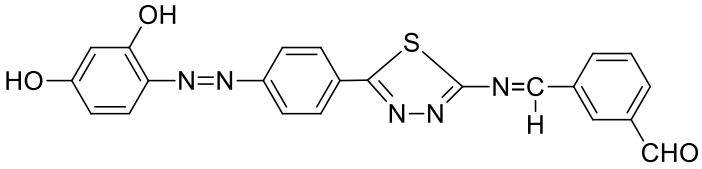
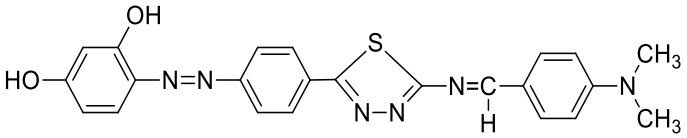
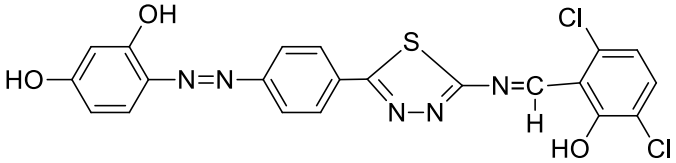
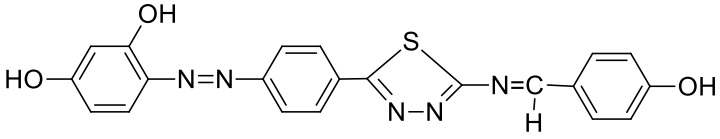
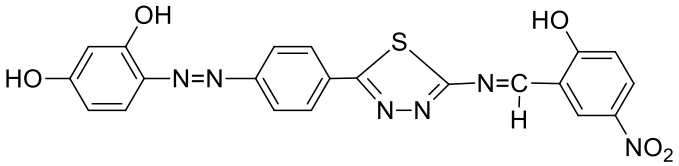
Comp. NO	Compound structure	Compound name

Table (2-1) : Chemical Structure and Name of Preparation Compounds [S1-S29]:

[S1]		2,4)4- , dihydroxyphenyl)diaze nyl)benzoic acid
[S2]		ethyl 4-((2,4-dihydroxyphenyl)diaze nyl)benzoate
[S3]		-2,4))-4 dihydroxyphenyl)diaze nyl)benzohydrazide
[S4]		-2,4))-4)-1 dihydroxyphenyl)diaze nyl)benzoyl)-1,2-dihydropyridazine-3,6-dione
[S5]		-2,4))-4)-2 dihydroxyphenyl)diaze nyl)benzoyl)-2,3- di hydro phthalazine-1,4-dione
[S6]		-2,4))-4)-2 dihydroxyphenyl)diaze nyl)cyclohexa-2,4-dienecarbonyl)-5-nitro-2,3-di hydro phthalazine-1,4-dione
[S7]		-2,4))-4)-1 dihydroxyphenyl)diaze nyl)benzoyl)tetrahydropyridazine-3,6-dione
[S8]		(4-((2,4-dihydroxyphenyl)diaze nyl)phenyl)(3,5- di methyl-1H-pyrazol -1-yl)methanone
[S9]		1-(4-((2,4-dihydroxyphenyl)diaze nyl)benzoyl)-3-methyl -1H-pyrazol -5(4H)-one

[S10]		(4-((2,4-dihydroxyphenyl)diazenyl)phenyl)(3-methyl-1H-pyrazol-1-yl)methanone
[S11]		1-(4-((2,4-dihydroxyphenyl)diazenyl)benzoyl)pyrazolidine-3,5-dione
[S12]		4))-4)- 5-amino -1,3,4-thiadiazol -2-yl)phenyl)diazenyl)benzene -1,3-diol
[S13]		4-((5-(4-((2,4-dihydroxyphenyl)diazenyl)phenyl)-1,3,4-thiadiazol -2-yl)diazenyl)-5-methy lbenzene-1,3-diol
[S14]		4-((4-(5-((2,5-dihydroxyphenyl)diazenyl) -1,3,4-thiadiazol -2-yl)phenyl)diazenyl)benzene -1,3-diol
[S15]		4-((4-(5-((2,4-dihy droxyphenyl)diazenyl)-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)benzene-1,3-diol
[S16]		3-((5-(4-((2,4-di hydroxyl phenyl)diazenyl)phenyl)-1,3,4-thiadiazol-2-yl)diazenyl)benzene-1,2-diol

[S17]		4-((4-(5-((4-hydroxy naphthalen -1-yl)diazenyl)-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)benzene-1,3-diol
[S18]		4-((4-(5-((2-hydroxy naphthalen-1-yl)diazenyl)-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)benzene-1,3-diol
[S19]		4-((4-(5-((Z)-(4-hydroxy-3-methyl naphthalen-1-yl)diazenyl)-1,3,4-thiadiazol -2-yl)phenyl)diazenyl)benzene -1,3-diol
[S20]		4-((4-(5-((3-hydroxy-2,4-dimethylphenyl)diazenyl)-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)benzene-1,3-diol
[S21]		4-((4-(5-((1H-1,2,4-triazol -5-yl)diazenyl)-1,3,4-thiadiazol-2-yl)phenyl)biszenyl)benzene -1,3-diol
[S22]		4-((4-(5-((3-mercapto -1H-1,2,4-triazol-5-yl)diazenyl)-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)benzene-1,3-diol
[S23]		4-((4-(5-((2-amino -5-methylphenyl)diazenyl)-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)benzene-1,3-diol

[S24]		))-5)-4))-4anthracen -9-ylmethylene)amino)-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)benzene -1,3-diol
[S25]		-2,4))-4)-5)))-3di hydroxyl phenyl) diazenyl)phenyl)-1,3,4-thiadiazol-2-yl)imino)methy l)benzaldehyde
[S26]		)-4))-5)-4))-4di methyl amino)benzylidene)amino)-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)benzene-1,3-diol
[S27]		-3,6))-5)-4))-4 dichloro -2-hydroxybenzylidene)amino)-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)benzene-1,3-diol
[S28]		-4))-5)-4))-4hydroxy benzylidene)amino)-1,3,4-thiadiazol -2-yl)phenyl)diazenyl)benzene -1,3-diol
[S29]		-2))-5)-4))-4hydroxy -5-nitrobenzylidene)amino)-1,3,4-thiadiazol-2-yl)phenyl)diazenyl)benzene-1,3-diol

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

Compound Number	M.P (^{0}C)	Yield (%)	Color	Molecular Weight (g\mole)	Molecular formula
[S1]	195-197	93	Orange	258	$C_{13}H_{10}N_2O_4$
[S2]	184-186	93	Orange	286	$C_{15}H_{14}N_2O_4$
[S3]	75-78	85	black	272	$C_{13}H_{12}N_4O_3$
[S4]	112-115	78	Light brown	352	$C_{17}H_{12}N_4O_5$
[S5]	91-93	88	Dark brown	402	$C_{21}H_{14}N_4O_5$
[S6]	120-122	81	Light brown	449	$C_{21}H_{15}N_5O_7$
[S7]	138-140	77	brown	354	$C_{17}H_{14}N_4O_5$
[S8]	120-122	82	Light brown	338	$C_{17}H_{14}N_4O_4$
[S9]	107-109	80	Dark brown	336	$C_{18}H_{16}N_4O_3$
[S10]	95-99	81	brown	322	$C_{17}H_{14}N_4O_3$
[S11]	111-113	76	Dark brown	350	$C_{19}H_{18}N_4O_3$
[S12]	180-182	78	Brown	313	$C_{14}H_{11}N_5O_2S_1$
[S13]	92-95	83	Dark brown	448	$C_{21}H_{16}N_6O_4S_1$
[S14]	90-93	85	Black	434	$C_{20}H_{14}N_6O_4S_1$
[S15]	165-168	86	Black	434	$C_{20}H_{14}N_6O_4S_1$
[S16]	180-182	87	Dark brown	434	$C_{20}H_{14}N_6O_4S_1$
[S17]	190-192	83	Light brown	468	$C_{24}H_{16}N_6O_3S_1$
[S18]	117-120	84	Brown	468	$C_{24}H_{16}N_6O_3S_1$
[S19]	95-98	87	Dark brown	482	$C_{25}H_{18}N_6O_3S_1$
[S20]	110-113	86	Light brown	446	$C_{22}H_{18}N_6O_3S_1$
[S21]	92-94	85	Dark brown	393	$C_{16}H_{11}N_9O_2S$
[S22]	91-93	88	brown	425	$C_{16}H_{11}N_9O_2S_2$
[S23]	88-90	77	Light	431	$C_{21}H_{17}N_7O_2S$

			brown		
[S24]	192-194	87	Light brown	501	$C_{29}H_{19}N_5O_2S$
[S25]	188-190	82	Light brown	429	$C_{22}H_{15}N_5O_3S_1$
2. **[S26]**	180-182	75	Dark brown	444	$C_{23}H_{20}N_6O_2S_1$
[S27]	183-186	86	Brown	486	$C_{21}H_{13}Cl_2N_5O_3S_1$
[S28]	190-192	80	black	417	$C_{21}H_{15}N_5O_3S_1$
[S29]	192-194	74	Light brown	462	$C_{21}H_{14}N_6O_5S_1$

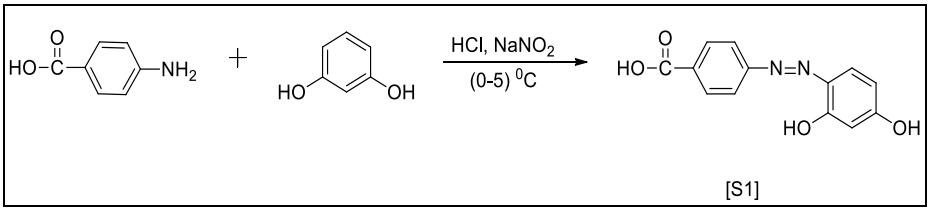
Chapter Three

Results and Discussion

3.Results and Discussion

3.1 Synthesis of Azo Compound [S1] :

The azo compound [S1] was synthesized by reaction of two organic compound ,a diazonium salt (*p*-amino benzoic acid) reacts as an electrophile with a coupling molecule that is rich in electrons (resorcinol) at (0-5 ^{0}C)



Equation 3-1: Synthesis of Compound [S1]

The FTIR spectrum exhibited absorption band at (1475) cm^{-1} for N=N and disappearance absorption band at (3458,3360) cm^{-1} for NH_2

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) Spectrum showed appearance signal at 6.53 for (H,OH) and signal at 10 for (H,COOH) .

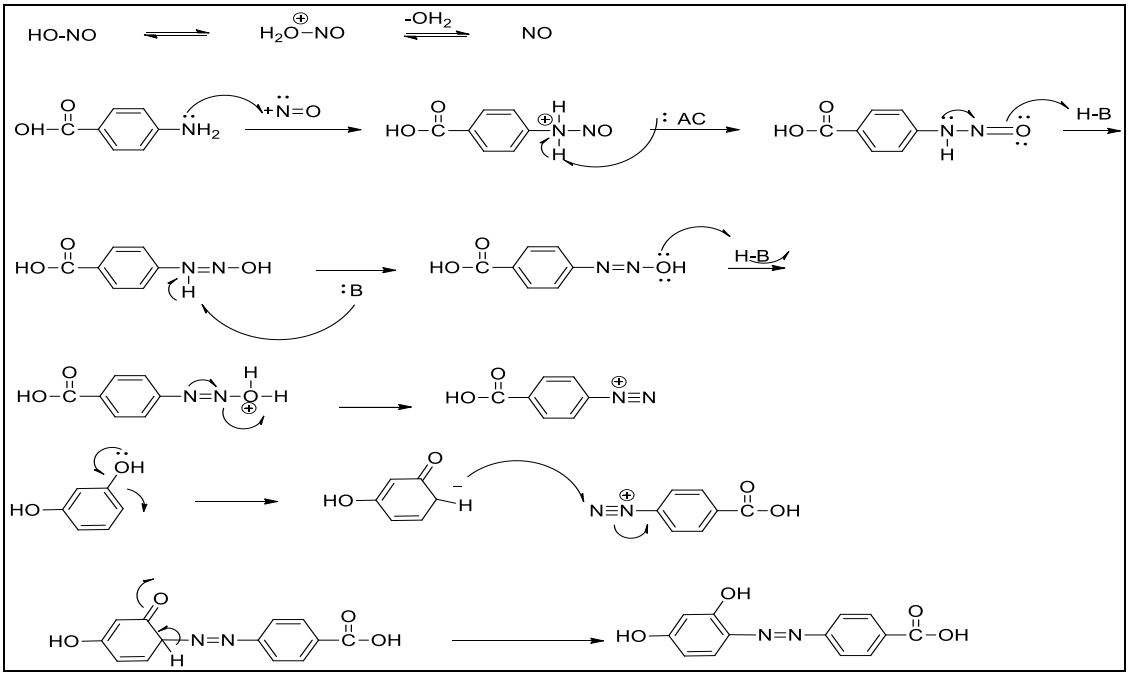
^{13}C-NMR (126 MHz,DMSO-d_6) :(δ, ppm) spectrum showed signals at 117-162 for Ar-C and signal at 169 for $C=O_{carboxylic\ acid}$

IR (*v*, cm^{-1}): O-H (3267),C-H $_{Ar}$ (3101), C=O $_{carboxylic\ acid}$ (1683), $C=C_{Ar}$ (1602), N=N (1475) ,C-O (1240)

^{1}H-NMR (δ, ppm): (7H,Ar-H) : 6.33-8.08 .

^{13}C-NMR (δ, ppm) : (117-162) for (12C,Ar-C),(169) for $C=O_{carboxylic\ acid}$.

The mechanism of synthesis azo is shown below[(94)]:



Scheme 3-1 : Mechanism of synthesis compound [S1]

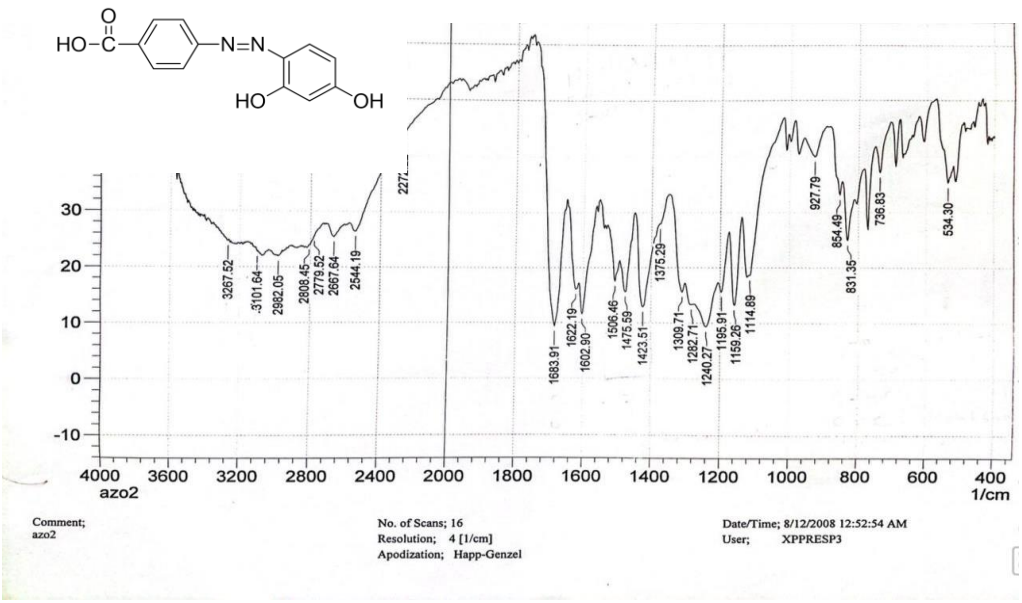


Figure 3-1 :FT-IR spectrum to compound [S1]

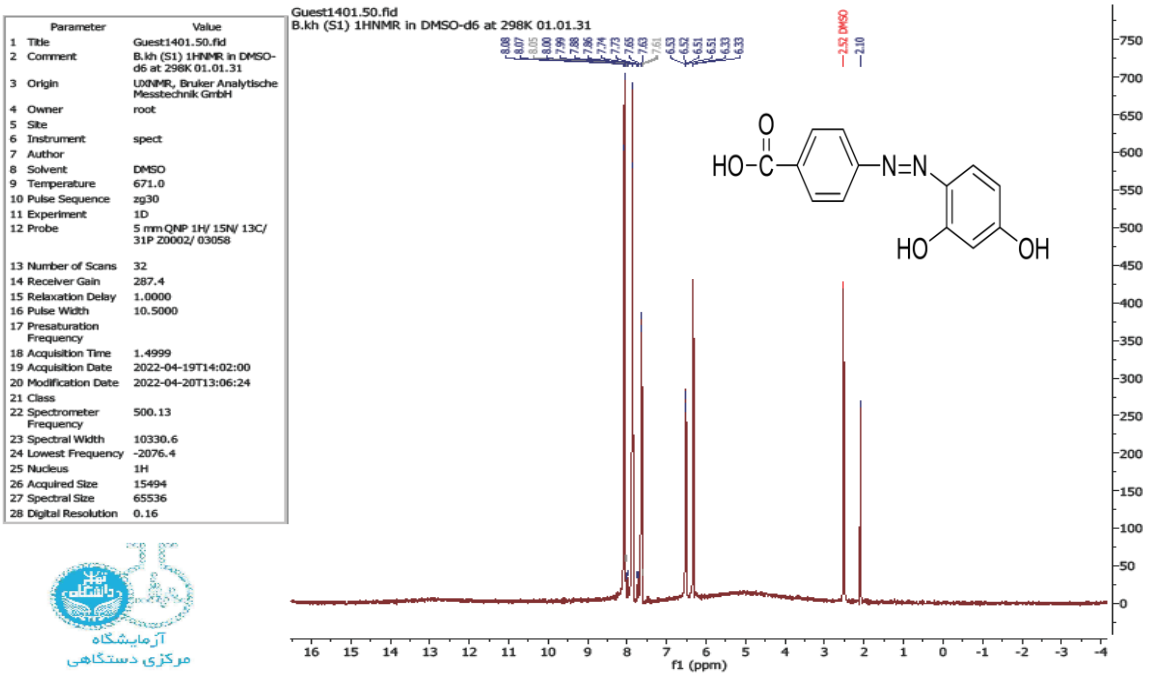


Figure 3-2: ^{1}H-NMR spectrum of compound [S1]

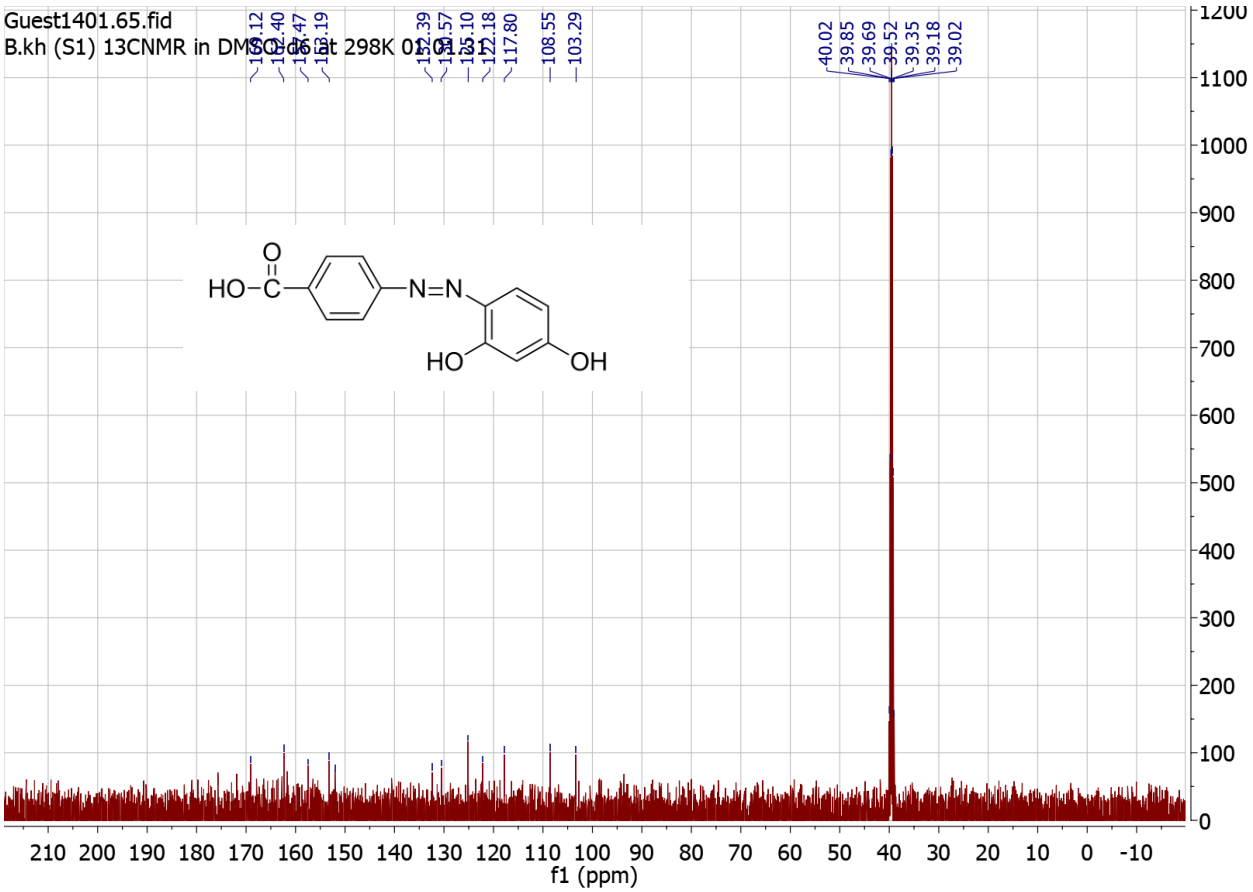
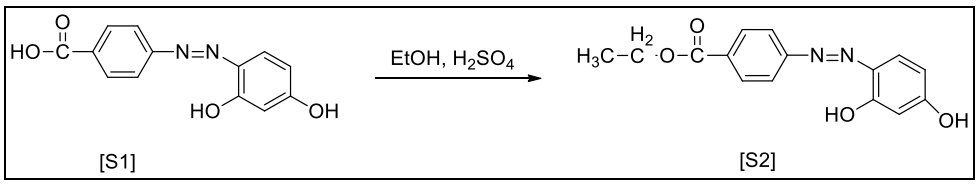


Figure 3-3 : ^{13}CNMR spectrum of compound [S1]

3.2 Synthesis of ester compound [S2] :

Ester compound was synthesized by Fischer esterification is an organic reaction that involved reaction of a carboxylic acid and an alcohol to give ester using an acid as catalyst ,usually concentrated sulfuric acid under reflux (overnight).



Equation 3-2: Synthesis of the Compound [S2]

The FTIR spectrum of compound [S2] exhibited absorption bands for C-H_{aliph} at (2937,2904) cm^{-1} and absorption band of C=O $_{ester}$ at (1716) cm^{-1} and disappearance of absorption band of C=O $_{carboxylic\ acid}$ at (1683) cm^{-1}

^{1}H-NMR (500MHz,DMSO-d_6) (δ, ppm): spectrum showed disappearance a signal at 10 for (H,OH) and appearance signal at 1.32 for (3H,CH_3), and at 1.36 for (2H,CH_2)

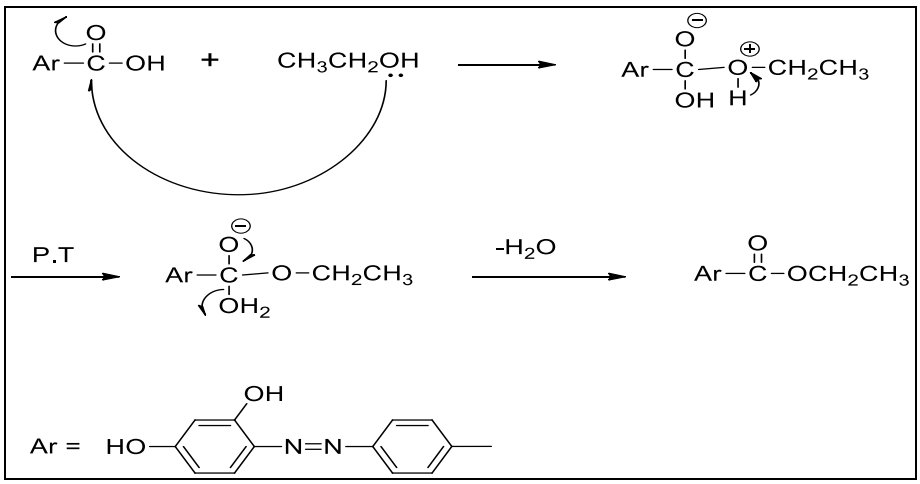
^{13}C-NMR (126 MHz,DMSO-d_6):(δ,ppm) spectrum showed signal at 14.16 for (CH_3) and signal at 39 for (CH_2) and signal at 60 for (C-O) and signals at 103-162 for(Ar-C)and signal at 165 for C=O_{ester}

IR (*v*,cm^{-1}): O-H (3410) ,C-H_{Ar} (2982) , C-H_{alph} (2937,2904) ,C=O_{ester} (1716) , C=C_{Ar} (1622), N=N (1516).

^{1}H-NMR(δ, ppm):(2H,CH_2,3H,CH_3): 1.32,1.36 ,(7H, Ar-H): 6.5-8.02 , (H,OH): 12.5

^{13}C-NMR (δ, ppm) : 14.16 for CH_3 , 39 for CH_2 , 60 for CH_2 ,103-162 for Ar-C , 165 for C=O_{ester}

The mechanism of synthesis ester is shown below [94]:



Scheme 3-2 : Mechanism of synthesis compound [S2]

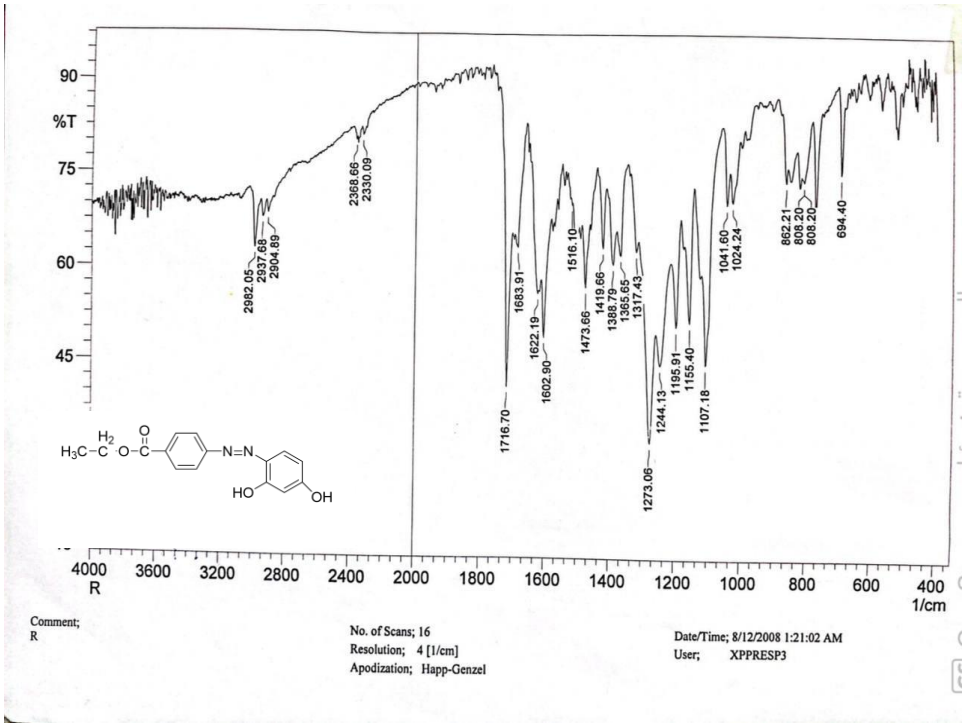
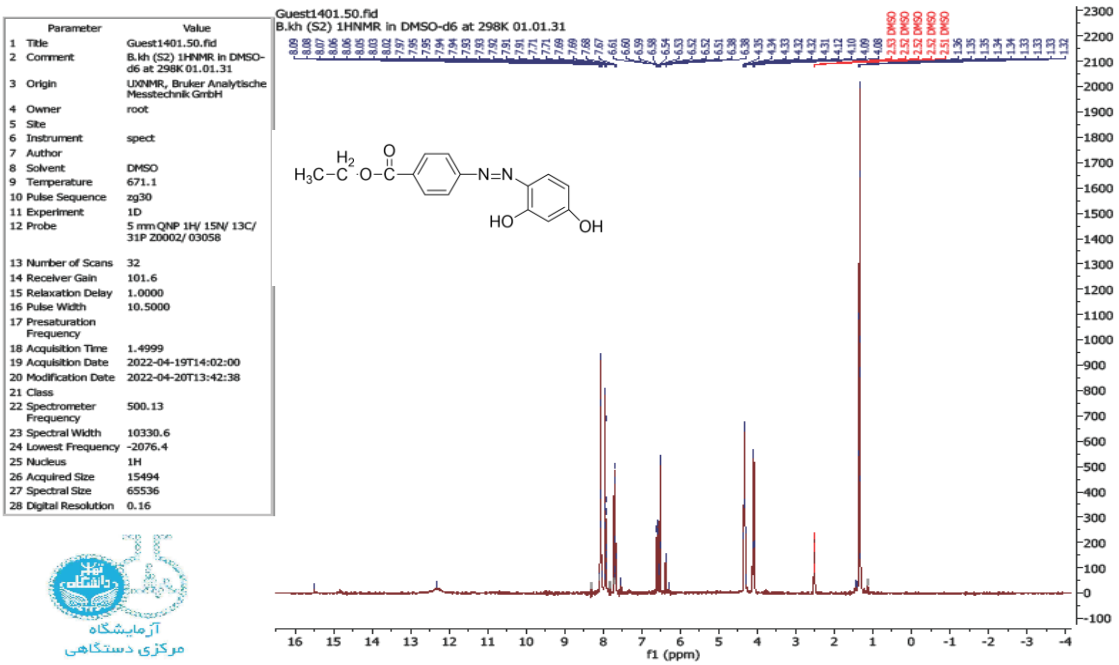


Figure 3-4: FT-IR spectrum of compound [S2]



	Parameter	Value
1	Title	Guest1401.50.fid
2	Comment	B.kh (S2) 1HNMR in DMSO-d6 at 298K 01.01.31
3	Origin	UXNMR, Bruker Analytische Messtechnik GmbH
4	Owner	root
5	Site	
6	Instrument	spect
7	Author	
8	Solvent	DMSO
9	Temperature	671.1
10	Pulse Sequence	zg30
11	Experiment	1D
12	Probe	5 mm QNP 1H/ 15N/ 13C/ 31P Z0002/ 03058
13	Number of Scans	32
14	Receiver Gain	101.6
15	Relaxation Delay	1.0000
16	Pulse Width	10.5000
17	Presaturation Frequency	
18	Acquisition Time	1.4999
19	Acquisition Date	2022-04-19T14:02:00
20	Modification Date	2022-04-20T13:42:38
21	Class	
22	Spectrometer Frequency	500.13
23	Spectral Width	10330.6
24	Lowest Frequency	-2076.4
25	Nucleus	1H
26	Acquired Size	15494
27	Spectral Size	65536
28	Digital Resolution	0.16

Figure 3-5 : ^{1}H-NMR spectrum of the compound [S2]

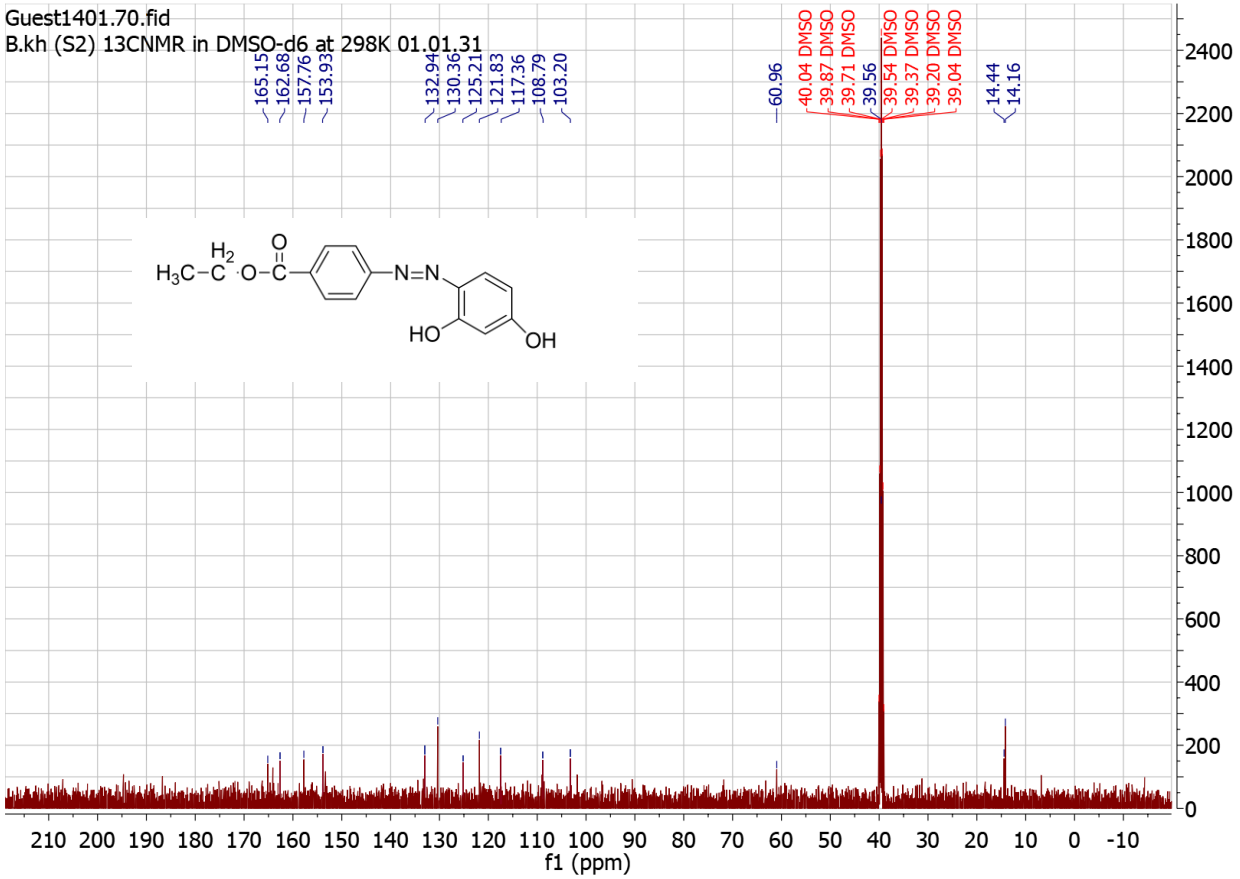
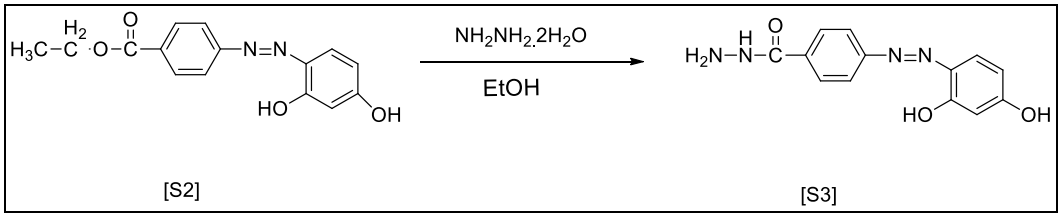


Figure 3-6 : ^{13}CNMR spectrum of the compound [S2]

3.3 Synthesis of Compound [S3] :

In this nucleophilic substitution reaction, hydrazine hydrate reacted as a nucleophile with carbonyl of ester in presence of absolute ethanol as solvent.



Equation 3-3: Synthesis of Compound [S3]

The FTIR spectrum showed disappearance absorption band at (1716) $cm^{-1}$for C=O ester and appearance absorption band at (1635) cm^{-1} for $C=O_{amide}$,and absorption band at (3346,3223) cm^{-1} for NH_2 absorption band at (3180) cm^{-1} for NH

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) spectrum showed a signals at 7.65 for protons of ($NH_{2)}$, signal at 5.96 for proton of (NH) and disappearance peak of protons of ester .

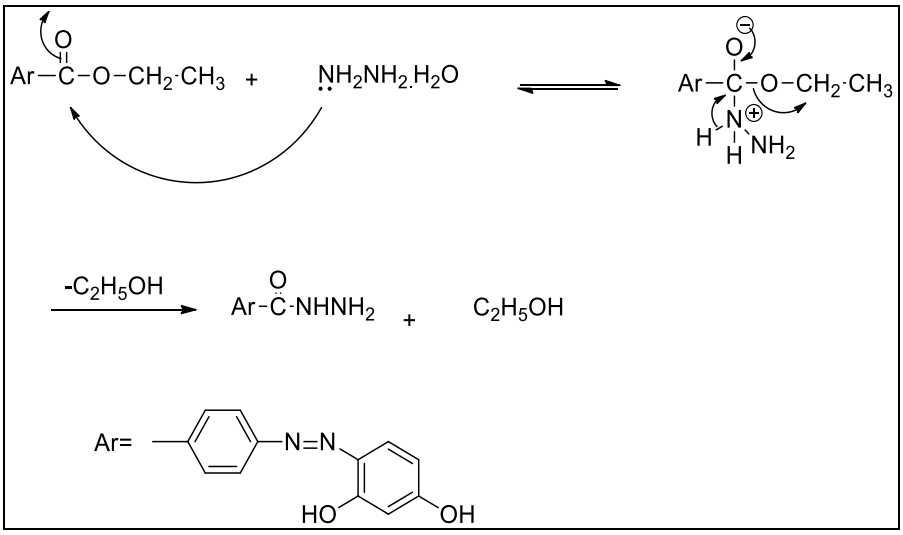
^{13}C-NMR (126 MHz,DMSO-d_6):(δ,ppm) spectrum showed disappearance signal of (C,CH_3) and signal of (C,CH_2) and appearance signals at 103-156 for Ar-C and signal at 165 for $C=O_{amide}$

IR (ν,cm^{-1}): OH (3423), NH_2 (3346,3223) , NH (3180),$C=O_{amide}$ (1685) $C=C_{Ar}$ (1653).,N=N(1599) .

^{1}H-NMR (δ, ppm): (H, NH) : 5.96, (H, NH_2) : 7.65 ,(H,OH):12.90 .

^{13}C-NMR (δ, ppm) :103-156 for (12C,Ar-C), 165 for ($C=O_{amide}$).

The mechanism of synthesis hydrazide is shown below [95]:



Scheme 3-3 : Mechanism of synthesis compound [S3]

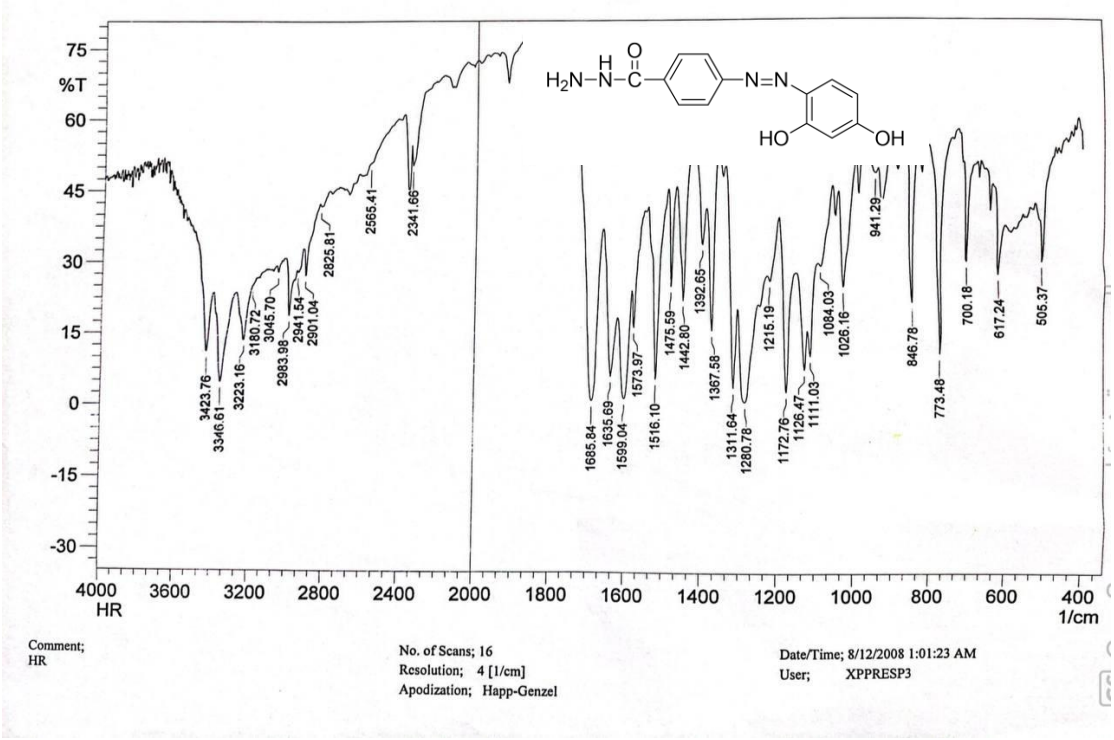


Figure 3-7: F.T-IR spectrum of compound[S3]

	Parameter	Value
1	Title	Guest1401.50.fid
2	Comment	B.kh (S3) 1HNMR in DMSO-d6 at 298K 01.01.31
3	Origin	UXNMR, Bruker Analytische Messtechnik GmbH
4	Owner	root
5	Site	
6	Instrument	spect
7	Author	
8	Solvent	DMSO
9	Temperature	671.1
10	Pulse Sequence	zg30
11	Experiment	1D
12	Probe	5 mm QNP 1H/ 15N/ 13C/ 31P Z0002/ 03058
13	Number of Scans	32
14	Receiver Gain	90.5
15	Relaxation Delay	1.0000
16	Pulse Width	10.5000
17	Presaturation Frequency	
18	Acquisition Time	1.4999
19	Acquisition Date	2022-04-19T14:02:00
20	Modification Date	2022-04-24T08:37:48
21	Class	
22	Spectrometer Frequency	500.13
23	Spectral Width	10330.6
24	Lowest Frequency	-2076.4
25	Nucleus	1H
26	Acquired Size	15494
27	Spectral Size	65536
28	Digital Resolution	0.16

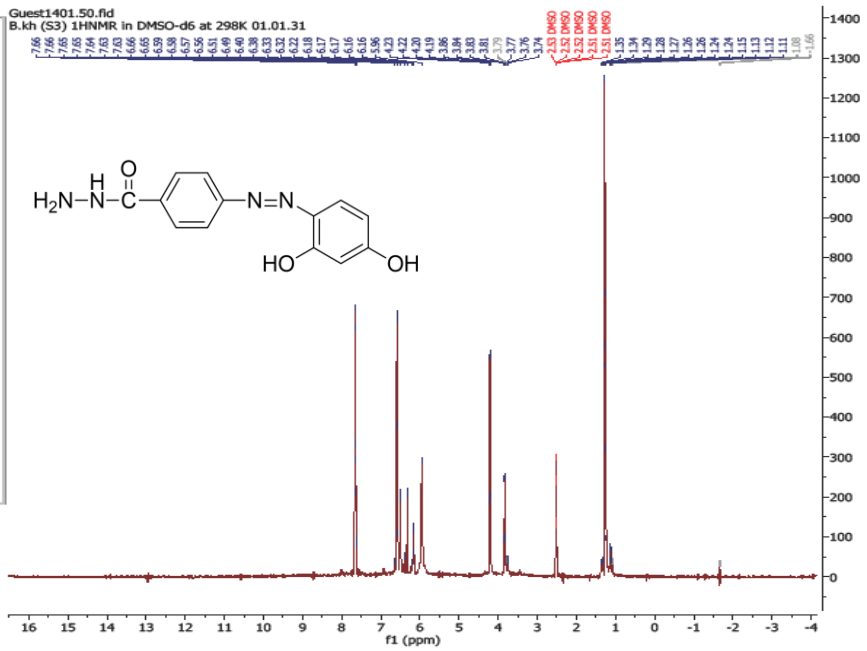


Figure3-8: ^{1}H-NMR spectrum of compound [S3]

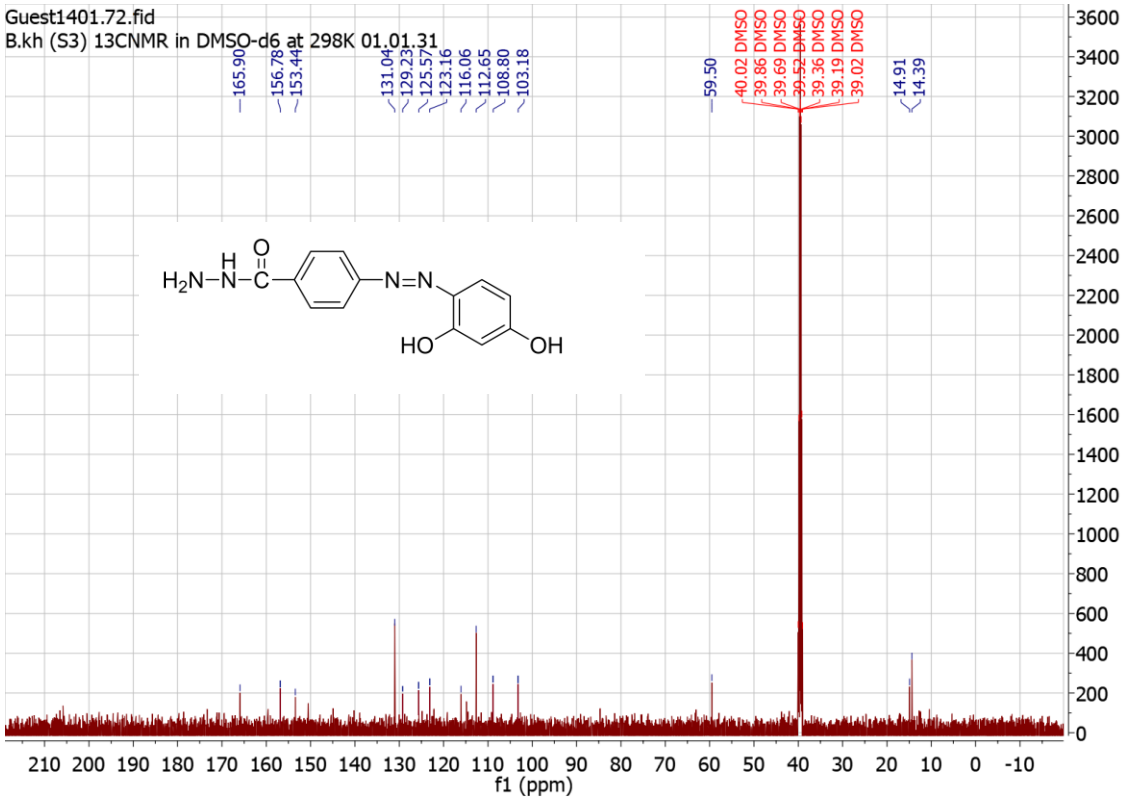
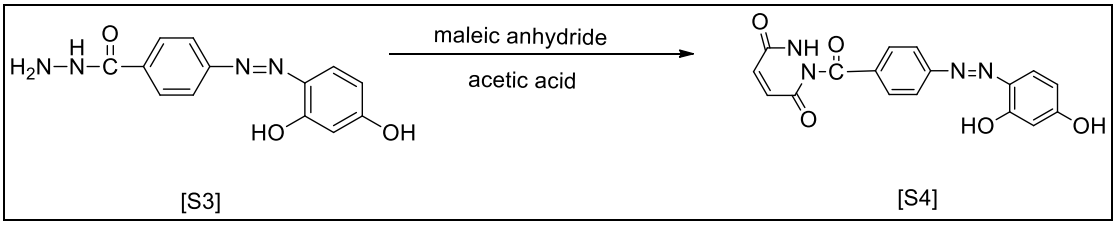


Figure 3-9 : ^{13}CNMR spectrum of compound [S3]

3.4 Synthesis of Compounds [S4-S7] :

Pyridazin-3,6-dione and phthalazin-3,8-dione derivatives were synthesized by the reaction of hydrazide derivatives [S3] with maleic anhydride ,phthalic anhydride,3-nitrophthalic anhydride and succinic anhydride on presence of acetic acid as catalyst and solvent, respectively.

Compound [S4]:



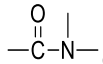
Equation 3-4: Synthesis of the Compound [S4]

The FTIR spectra of compound [S4] showed disappearance of NH_2 and NH bands in the region (3423,3346,3223) cm^{-1} and appearance band of (NH) at (3309) cm^{-1} and appearance a band of $C=O_{amide}$ at (1716) cm^{-1} .

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) Spectrum of compound [S4] showed appearance signal at 9.9 for (H,NH) and signal at 10.23 for (H,OH) and disappearance signal of (H,NH_2) .

^{13}C-NMR (126 MHz,DMSO-d_6):(δ,ppm) spectrum showed appearance signals at 117-136 for Ar-C and signal at 172 for $C=O_{amide}$

IR (*v*,cm^{-1}): OH(3400) ,NH (3309),C-H_{Ar} (3050) , C-$H_{alipatic}$ (2980) ,C=O (1716) ,C=C (1639),N=N (1604) .

^{1}H-NMR (δ, ppm) : (9H,Ar-H) :6.15-7.73 , (H-NH) : 9.9 ,(H, OH) : 10.23 . ^{13}C-NMR (δ, ppm) : 117-136 for Ar-C, 172 for  amide.

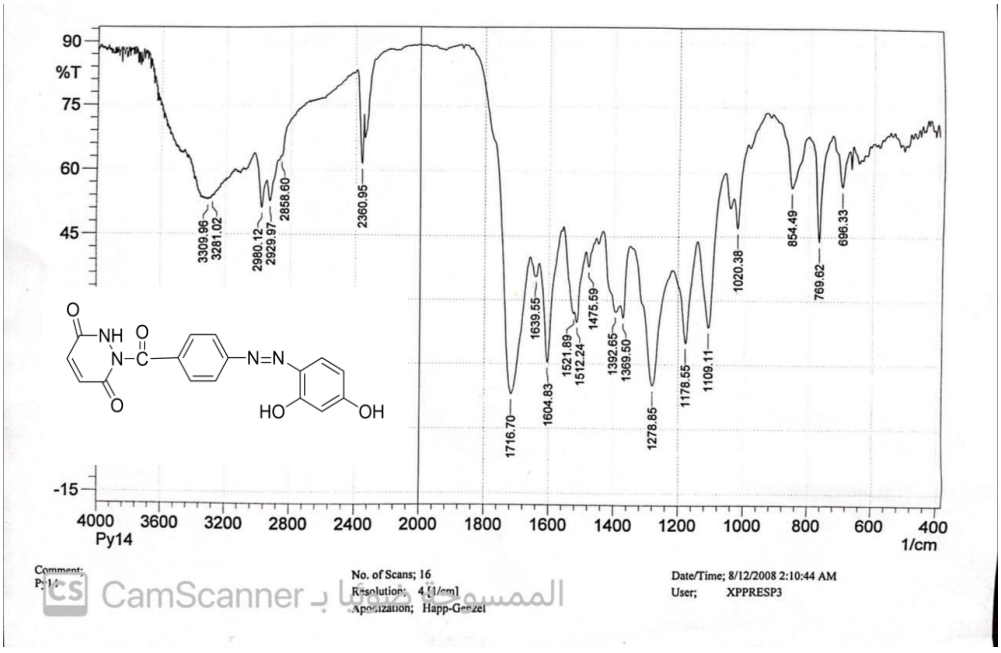
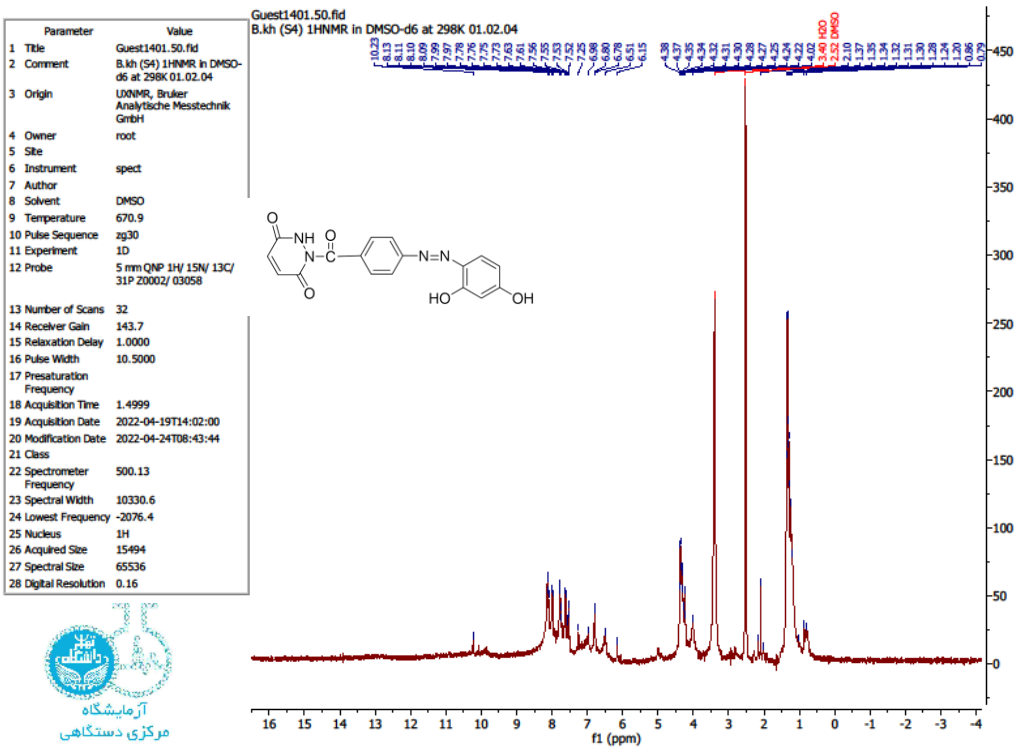


Figure3-10: F.T-IR spectrum of compound [S4]



Parameter	Value
1 Title	Guest1401.50.fid
2 Comment	B.kh (S4) 1HNMR in DMSO-d6 at 298K 01.02.04
3 Origin	UXNMR, Bruker Analytische Messtechnik GmbH
4 Owner	root
5 Site	
6 Instrument	spect
7 Author	
8 Solvent	DMSO
9 Temperature	670.9
10 Pulse Sequence	zg30
11 Experiment	1D
12 Probe	5 mm QNP 1H/ 15N/ 13C/ 31P Z0002/ 03058
13 Number of Scans	32
14 Receiver Gain	143.7
15 Relaxation Delay	1.0000
16 Pulse Width	10.5000
17 Presaturation Frequency	
18 Acquisition Time	1.4999
19 Acquisition Date	2022-04-19T14:02:00
20 Modification Date	2022-04-24T08:43:44
21 Class	
22 Spectrometer Frequency	500.13
23 Spectral Width	10330.6
24 Lowest Frequency	-2076.4
25 Nucleus	1H
26 Acquired Size	15494
27 Spectral Size	65536
28 Digital Resolution	0.16

Figure 3-11: ^{1}H-NMR spectrum of compound [S4]

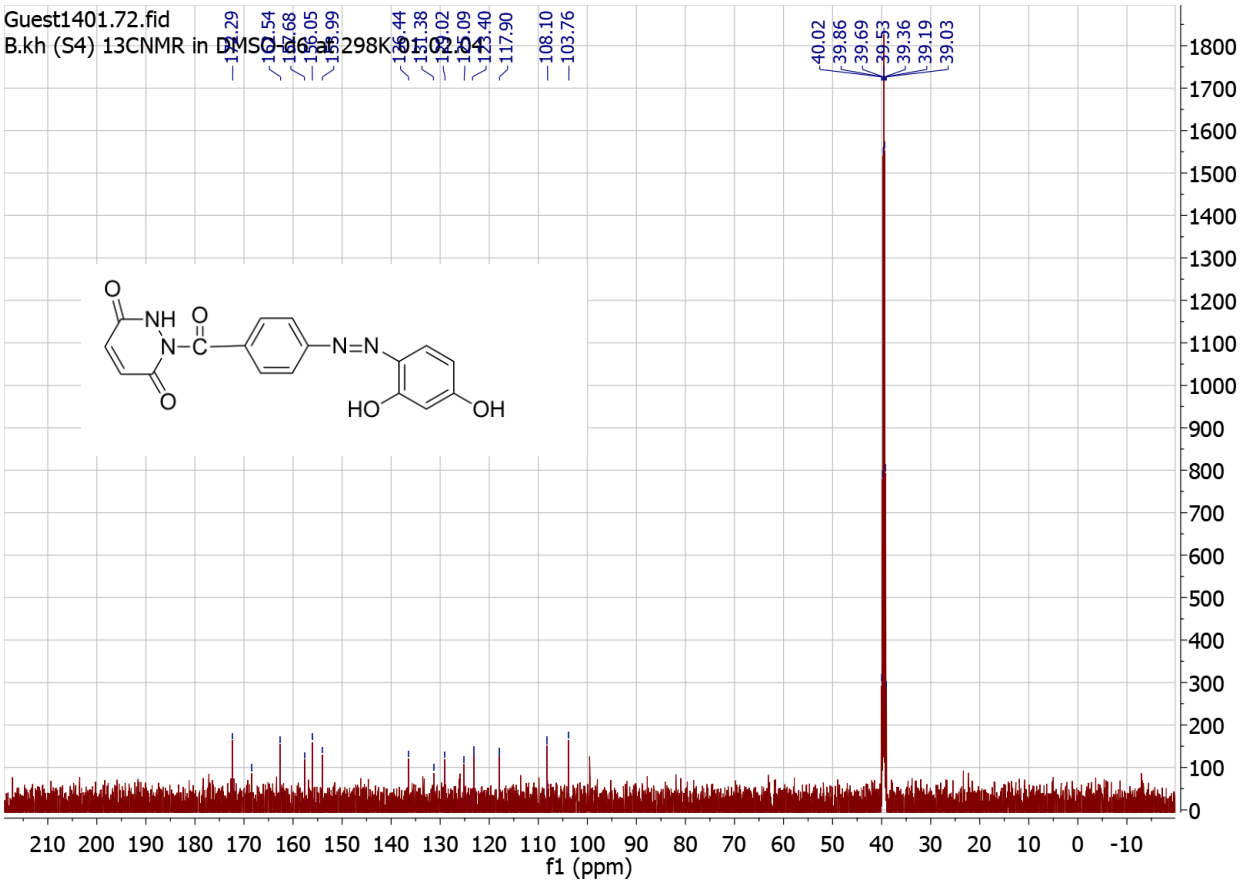
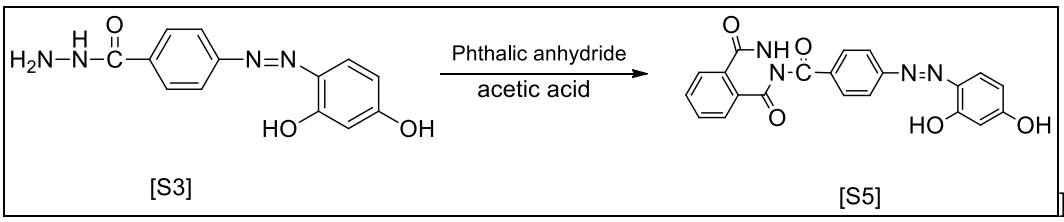


Figure 3-12: ^{13}CNMR spectrum of compound [S4]

Compound [S5]:



Equation 3-5: Synthesis of the Compound [S5]

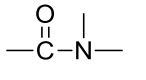
The FTIR spectra of compound [S5] showed disappearance of NH_2 and NH bands in the region (3423,3346,3223) cm^{-1} and appearance band of (NH) at (3317) cm^{-1} and appearance a band of $C=O_{amide}$ at (1708) cm^{-1} .

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) Spectrum of compound [S5] showed appearance signal at 9.33 for (H,NH) and disappearance signal of (H,NH_2) .

^{13}C-NMR (126 MHz,DMSO-d_6):(δ,ppm) spectrum showed appearance signals at 123-136 for Ar-C and signal at 172 for $C{=}O_{amide}$

IR (ν,cm^{-1}) :OH(3400), NH (3317) , C-H_{Ar} (3082) ,C=O (1708),C=C (1608),N=N (1606).

^{1}H-NMR (δ, ppm) : ,(H,NH):9.33 ,(11 H,Ar-H) :6.49 -8.13,(H,OH):10.3 .

^{13}C-NMR (δ, ppm) : 123 – 136 for C=C_{Ar} ,172 for  amide

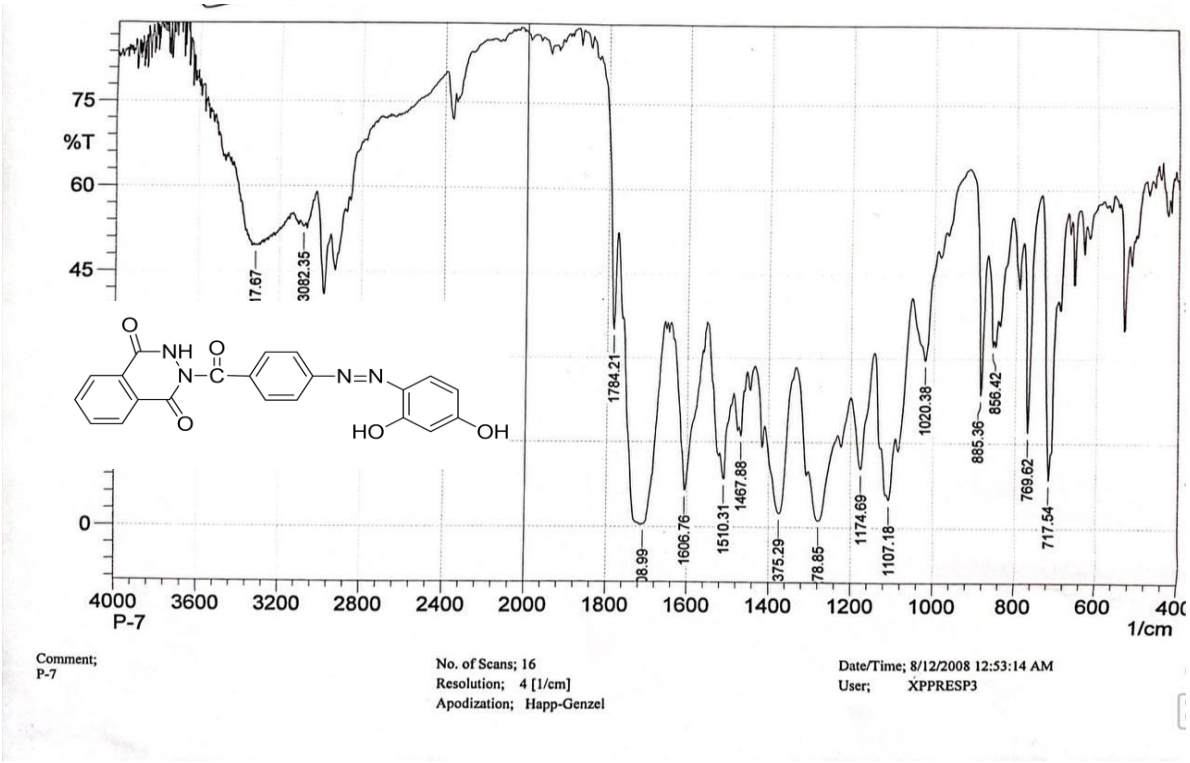


Figure 3-13: F.T-IR spectrum of compound [S5]

Parameter		Value
1	Title	Guest1401.50.fid
2	Comment	B.kh (S5) 1HNMR in DMSO-d6 at 298K 01.02.04
3	Origin	UXNMR, Bruker Analytische Messtechnik GmbH
4	Owner	root
5	Site	
6	Instrument	spect
7	Author	
8	Solvent	DMSO
9	Temperature	671.1
10	Pulse Sequence	zg30
11	Experiment	1D
12	Probe	5 mm QNP 1H/ 15N/ 13C/ 31P Z0002/ 03058
13	Number of Scans	32
14	Receiver Gain	128.0
15	Relaxation Delay	1.0000
16	Pulse Width	10.5000
17	Presaturation Frequency	
18	Acquisition Time	1.4999
19	Acquisition Date	2022-04-19T14:02:00
20	Modification Date	2022-04-24T09:22:14
21	Class	
22	Spectrometer Frequency	500.13
23	Spectral Width	10330.6
24	Lowest Frequency	-2076.4
25	Nucleus	1H
26	Acquired Size	15494
27	Spectral Size	65536
28	Digital Resolution	0.16

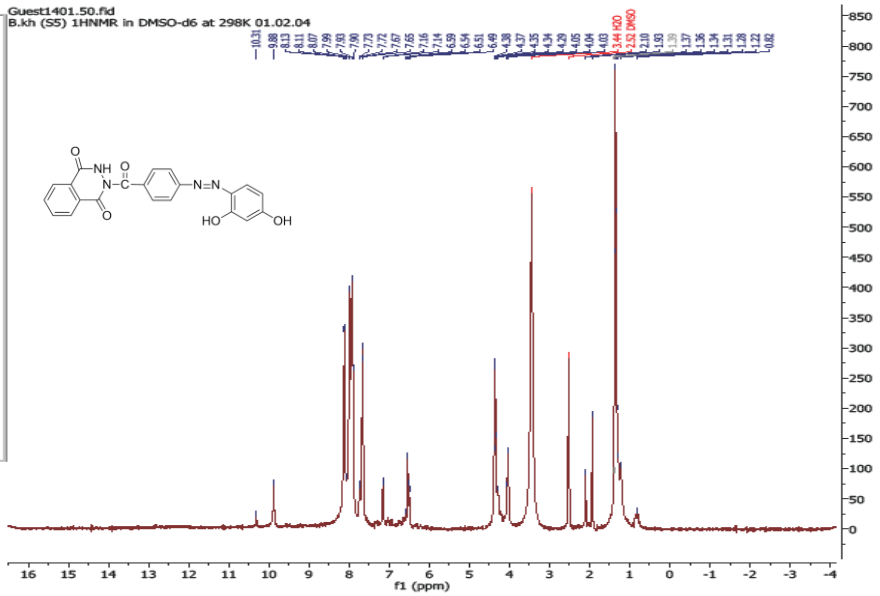


Figure 3-14: ^{1}H-NMR spectrum of compound [S5]

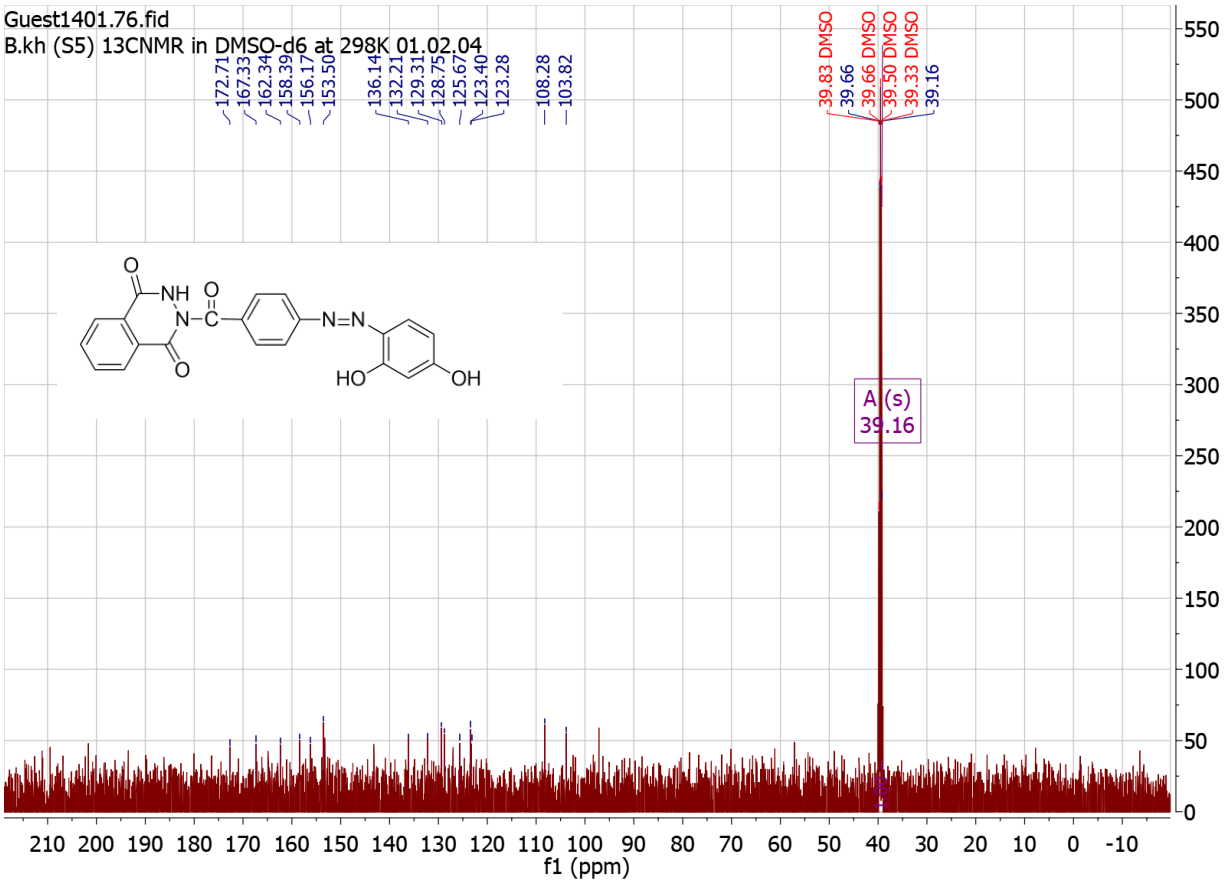
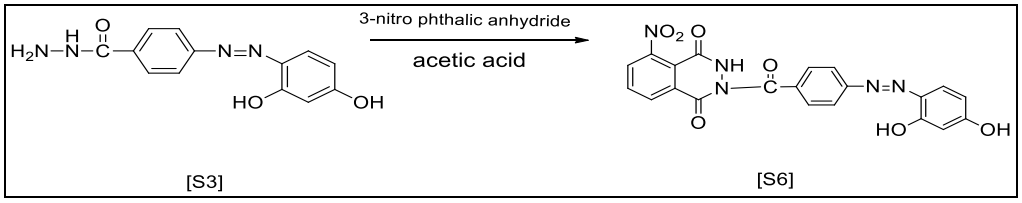


Figure3-15: ^{13}CNMR spectrum of compound [S5]

Compound [S6]:



Equation 3-6: Synthesis of the Compound [S6]

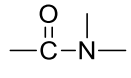
The FTIR spectra of compound [S6] show disappearance of NH_2 and NH bands in the region (3423,3346,3223) cm^{-1} and appearance band of (NH) at (3090) cm^{-1} and appearance a band of $C=O_{amide}$ at (1732) cm^{-1} .

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) Spectrum of compound [S6] show appearance signal at 9.5 for (H,NH) and disappearance signal of (H,NH_2) .

^{13}C-NMR (126 MHz,DMSO-d_6):(δ,ppm) spectrum show appearance signals at 118-145 for Ar-C and signal at 165 for $C{=}O_{amide}$

IR (ν,cm^{-1}): NH(3090) ,C-H_{Ar} (2982) ,C=O (1732) ,C=C (1606) N=N (1581) ,NO_2 (1541).

^{1}H-NMR (δ, ppm) : .(10H,Ar-H) :6.50 -8.37, (H-NH): 9.5,(H,OH):10.20 .

^{13}C-NMR (δ, ppm) :118-145 for Ar-C, 165 for  amide

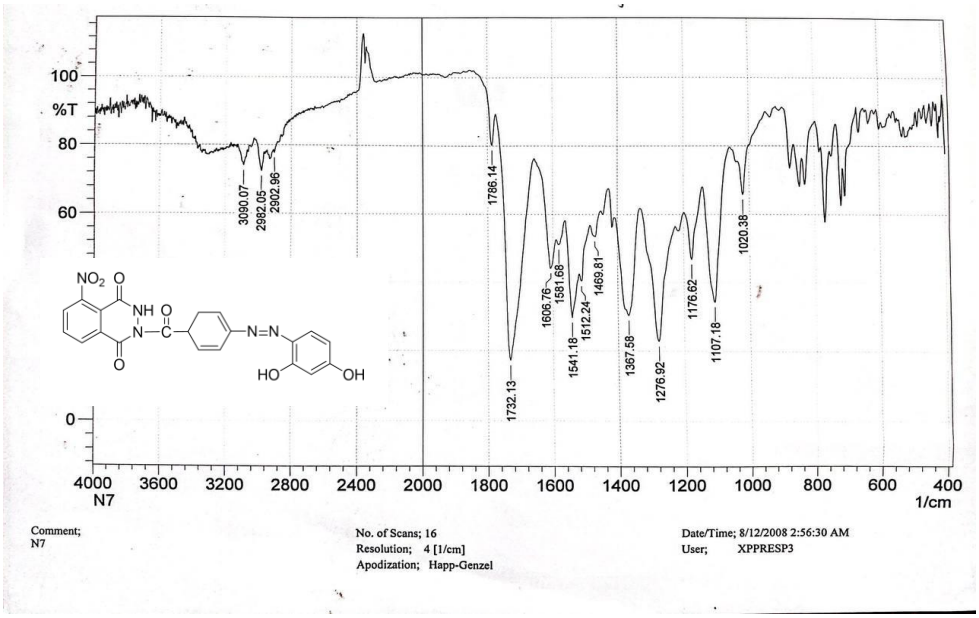


Figure3-16: F.T-IR spectrum of compound [S6]

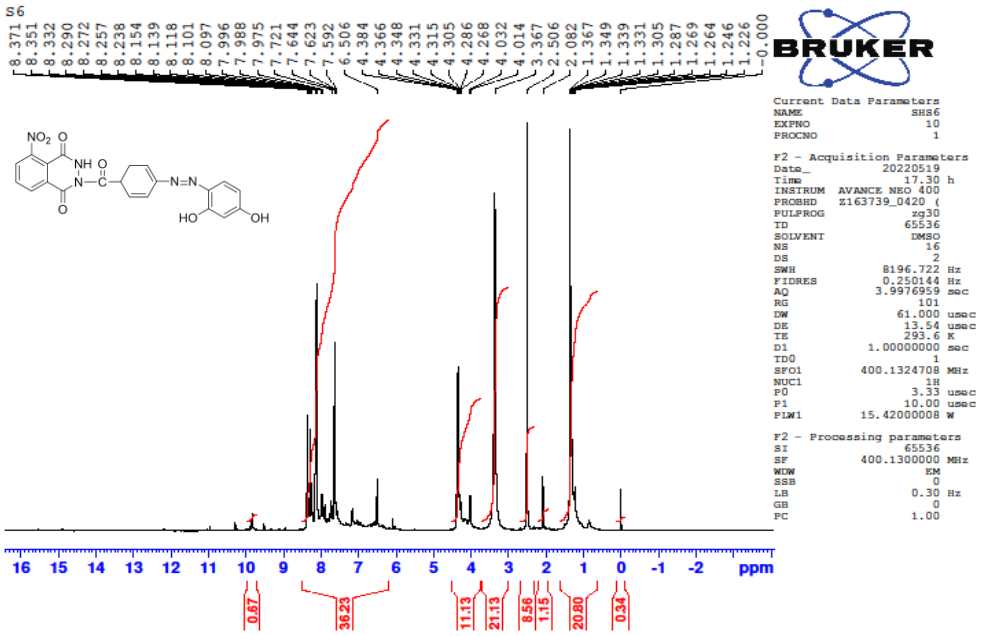


Figure 3-17: ^{1}H-NMR spectrum of compound [S6]

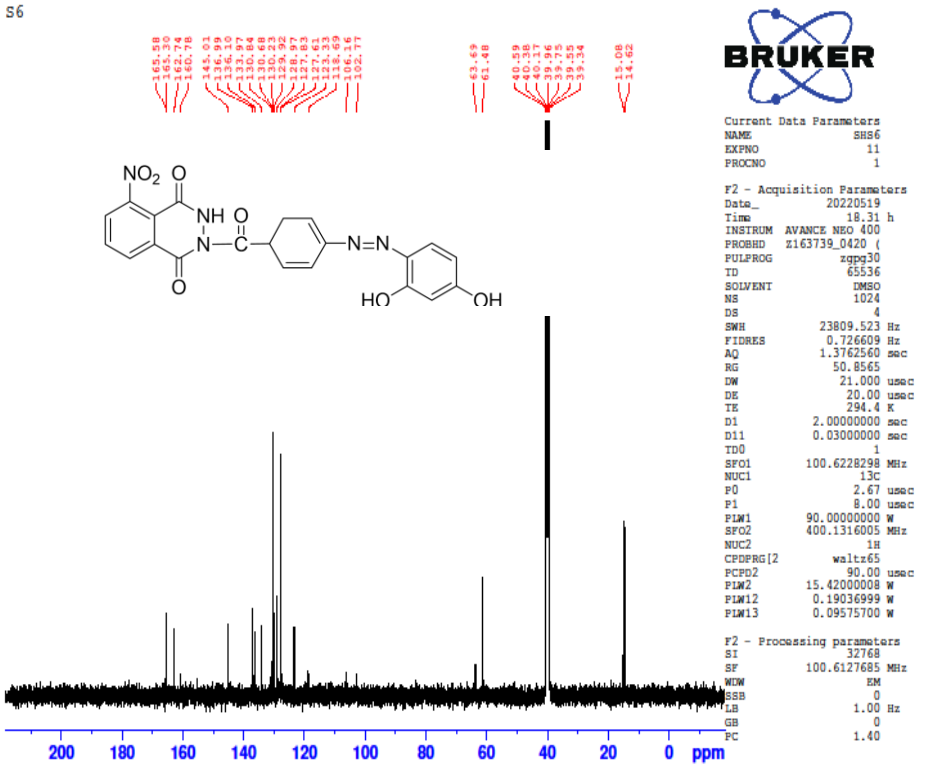
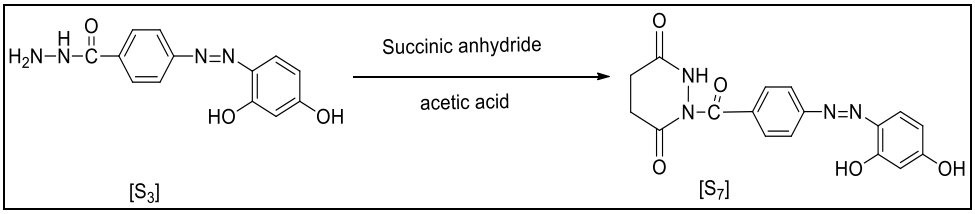


Figure3-18: ^{13}CNMR spectrum of compound [S6]

Compound [S7]:



Equation 3-7: Synthesis of the Compound [S7]

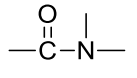
The FTIR spectra of compound [S7] show disappearance of NH_2 and NH bands in the region (3423,3346,3223) cm^{-1} and appearance band of (NH) at (3221) cm^{-1} and appearance a band of $C=O_{amide}$ at (1707) cm^{-1} .

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) Spectrum of compound [S7] show appearance signal at 9.5 for (H,NH) and disappearance signal of (H,NH_2) .

^{13}C-NMR (126 MHz,DMSO-d_6):(δ,ppm) spectrum show appearance signals at 103-136 for Ar-C and signal at 174 for $C{=}O_{amide}$

IR (ν,cm^{-1}): OH(3329) ,NH(3221) ,C-H_{Ar} (3076) , C-$H_{aliphatic}$ (2980),C=O (1707) ,C=C (1600) ,N=N (1575).

^{1}H-NMR (δ, ppm) : (7H,Ar-H) : 7.03-7.99 ,(H,NH) : 9.5,(H,OH): 10.5 .

^{13}C-NMR (δ, ppm) :103 -136 for Ar-C ,174 for  amide

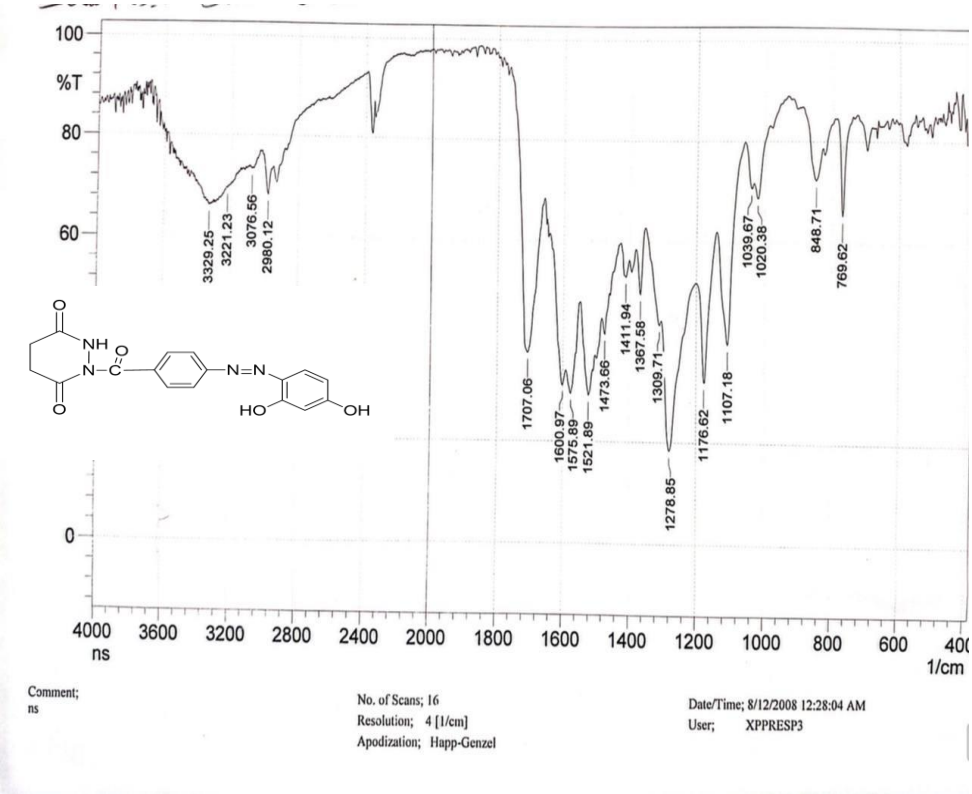


Figure3-19: F.T-IR spectrum of compound [S7]

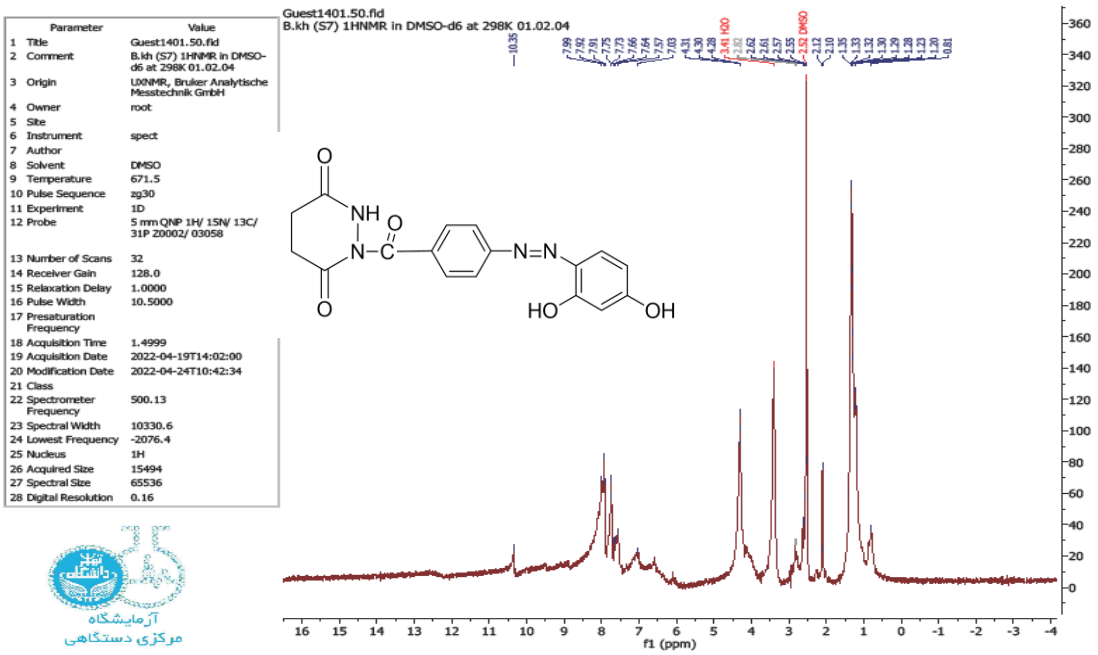


Figure3-20: ^{1}H-NMR spectrum of compound [S7]

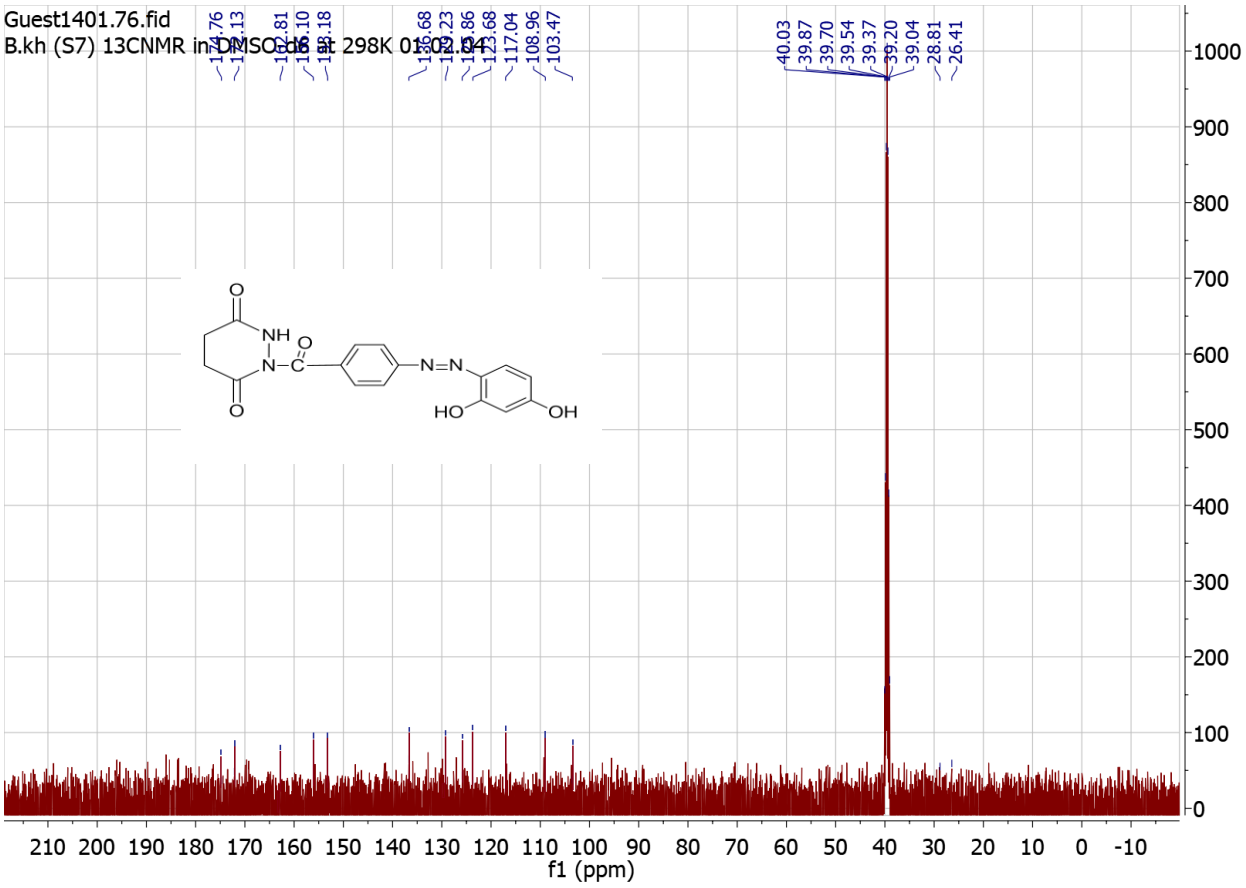
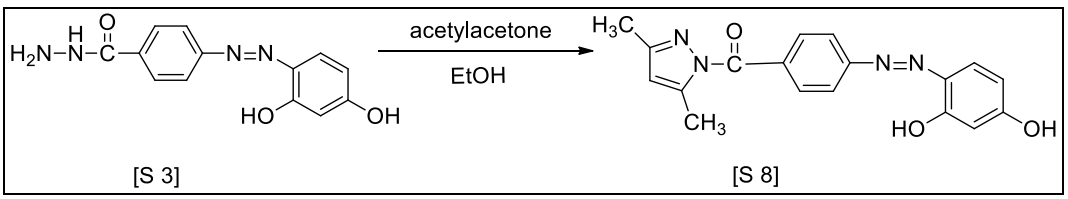


Figure3-21: ^{13}CNMR spectrum of compound [S7]

3.5 Synthesis of Compounds [S8-S11] :

The pyrazole derivatives were prepared through the reaction of hydrazide derivatives [S3] with (acetyl acetone, ethyl acetoacetate, methyl acetoacetate ,diethylmalonate) respectively.

Compound [S8]:



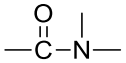
Equation 3-8: Synthesis of Compound[S8]

The FTIR spectra of compound [S8] show disappearance of NH_2 and NH bands in the region (3423,3346,3223) cm^{-1} and appearance of $C=O_{amide}$ band at (1705)cm^{-1}. and appearance of C-H aliphatic at (2928) cm^{-1} and C=C aromatic at (1519) cm^{-1}. ^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) Spectrum of compound [S8] show appearance signal at 1.37 for (H,CH_3) and disappearance signal of (H,NH_2) and signal of (H,NH)

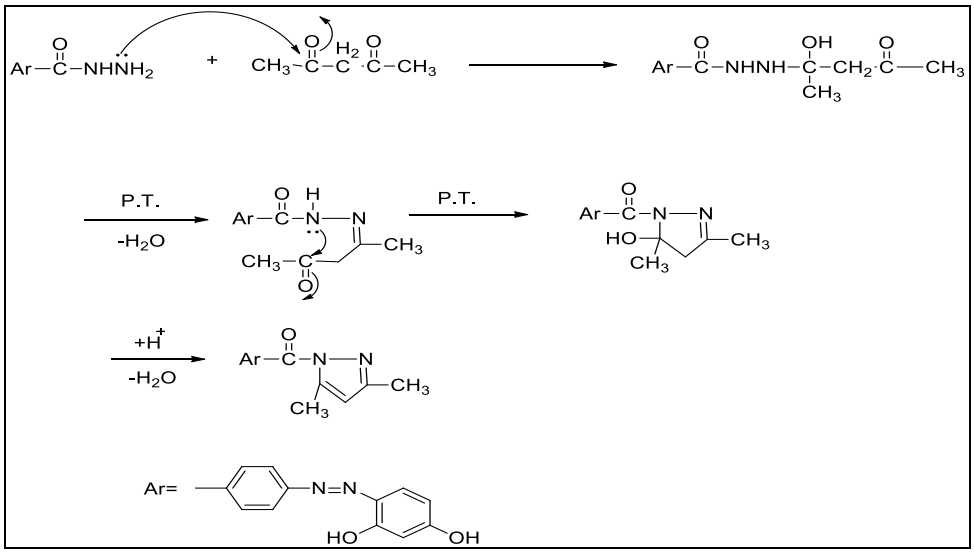
^{13}C-NMR (126 MHz,DMSO-d_6):(δ,ppm) spectrum show appearance signal of ($2CH_3$) and signals at 117-130 for Ar-C and signal at 170 for $C=O_{amide}$

IR (ν,cm^{-1}) :O-H (3350) ,C-H_{Ar} (3024) ,C-H $_{aliph}$ (2928) ,C=O (1705),C=N (1602) , $C=C_{Ar}$ (1519) ,N=N (1575) .

^{1}H-NMR (δ, ppm): ($6H,CH_3$) : 1.37 , (7H,Ar-H) : 7.02-8.15

^{13}C-NMR (δ, ppm) : 18.21 for CH_3 ,117-130 for Ar-C ,170 for  amide.

The mechanism of synthesis pyrazole is shown below [96]:



Scheme 3-4 Mechanism of synthesis compound [S8]

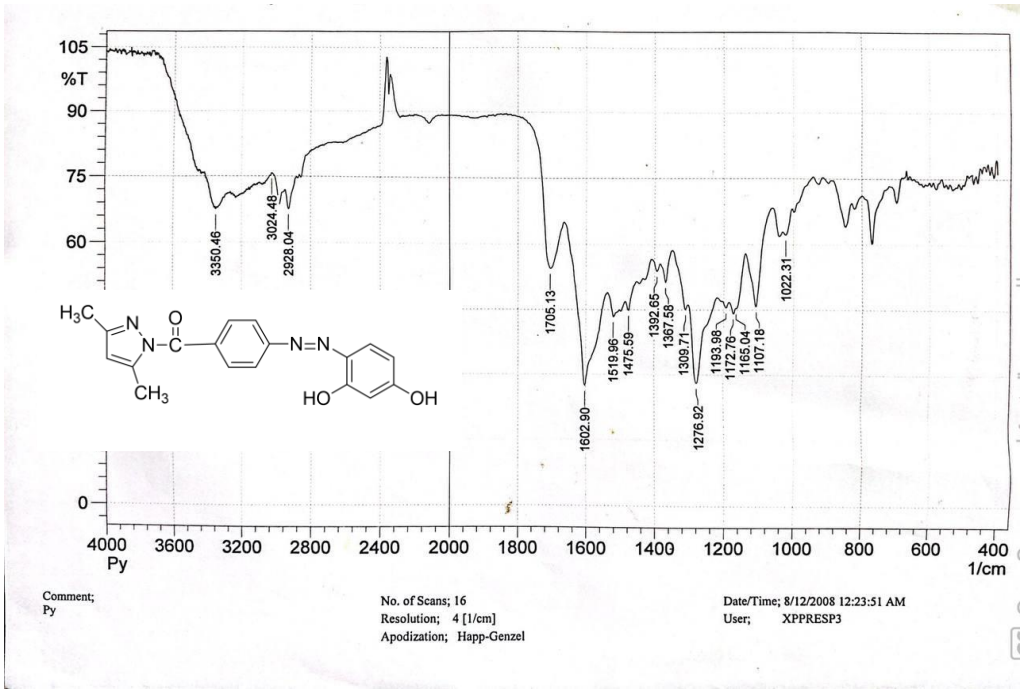


Figure3-22: F.T-IR spectrum of compound [S8]

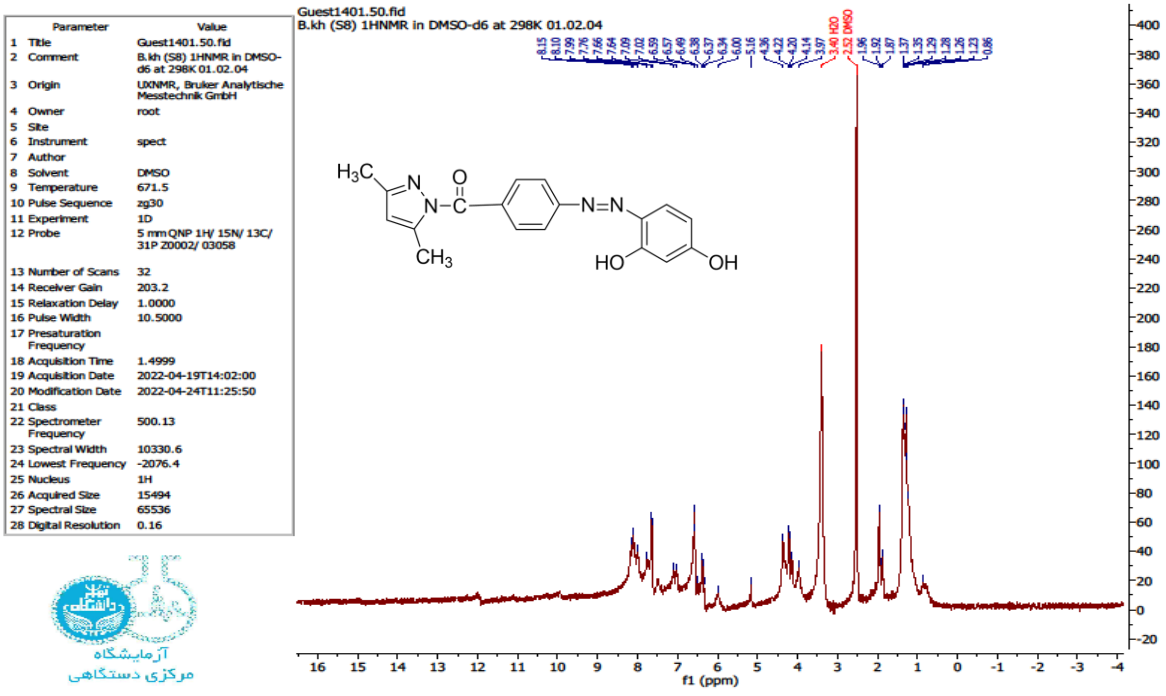


Figure 3-23: ^{1}H-NMR spectrum of compound [S8]

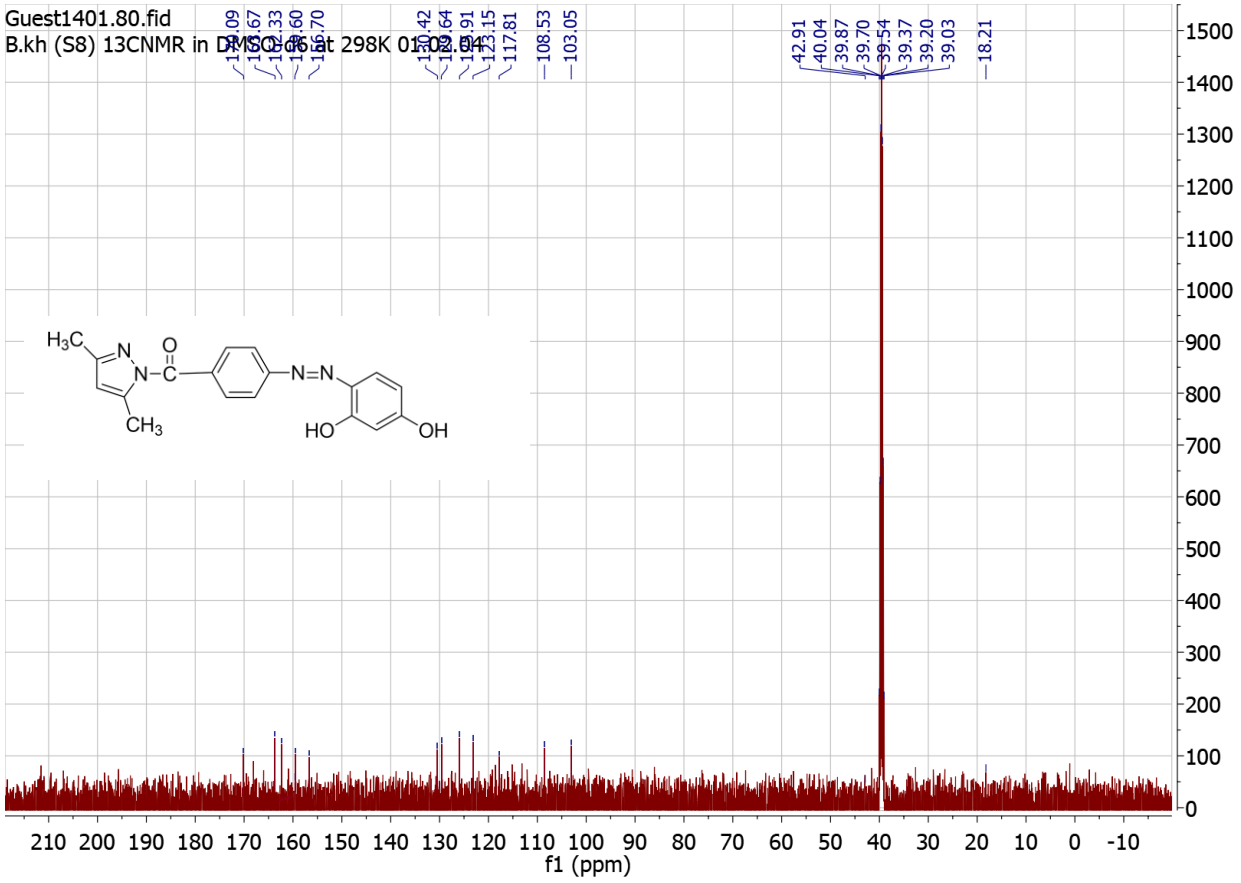
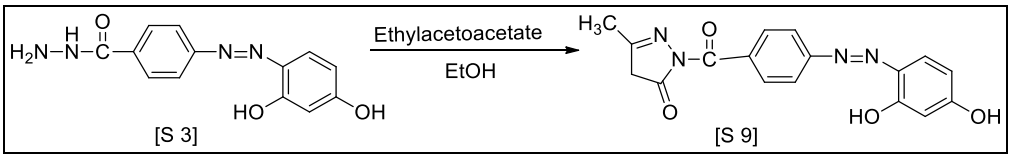


Figure3-24: ^{13}CNMR spectrum of compound [S8]

Compound [S9] :



Equation 3-9: Synthesis of Compound[S9]

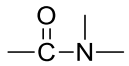
The FTIR spectra of compound [S9] show disappearance of NH_2 and NH bands in the region (3423,3346,3223) cm^{-1} and appearance of $C=O_{amide}$ band at (1699)cm^{-1}. and appearance of C-H aliphatic at (2980) cm^{-1} and C=C aromatic at (1577) cm^{-1}.

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) Spectrum of compound [S9] show appearance signal at 1.34 for (H,CH_3) and disappearance signal of (H,NH_2) and signal of (H,NH)

^{13}C-NMR (126MHz,DMSO-d_6):(δ,ppm) spectrum show appearance signals at 117-132 for Ar-C and signal at 165 for C=O_{amide}

IR (*v*,cm^{-1}): O-H (3348) ,C-H_{Ar} (3057) ,C-H $_{aliph}$ (2980) ,C=O (1699) ,C=N (1602), C=C_{Ar} (1577) ,N=N (1521).

^{1}H-NMR (δ, ppm) : ,(3H,CH_3): 1.34 ,(7H,Ar-H) :6.31-8.11

^{13}C-NMR (δ, ppm) : 117-132 for Ar-C , 165 for  amide

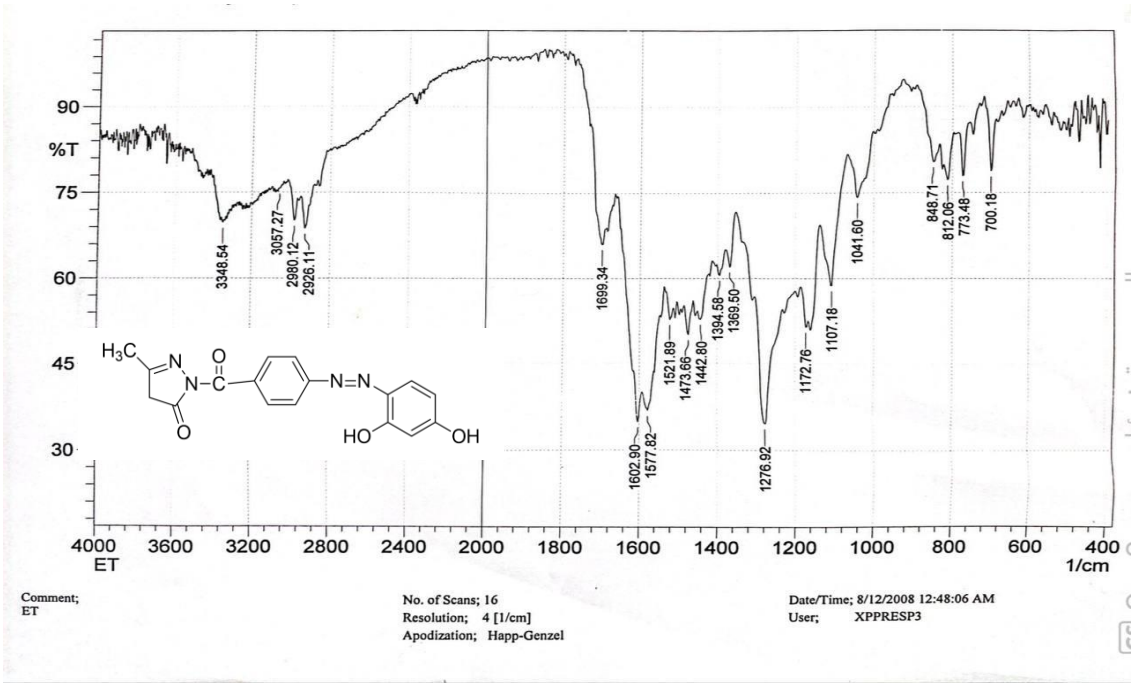


Figure3-25: F.T-IR spectrum of compound [S9]

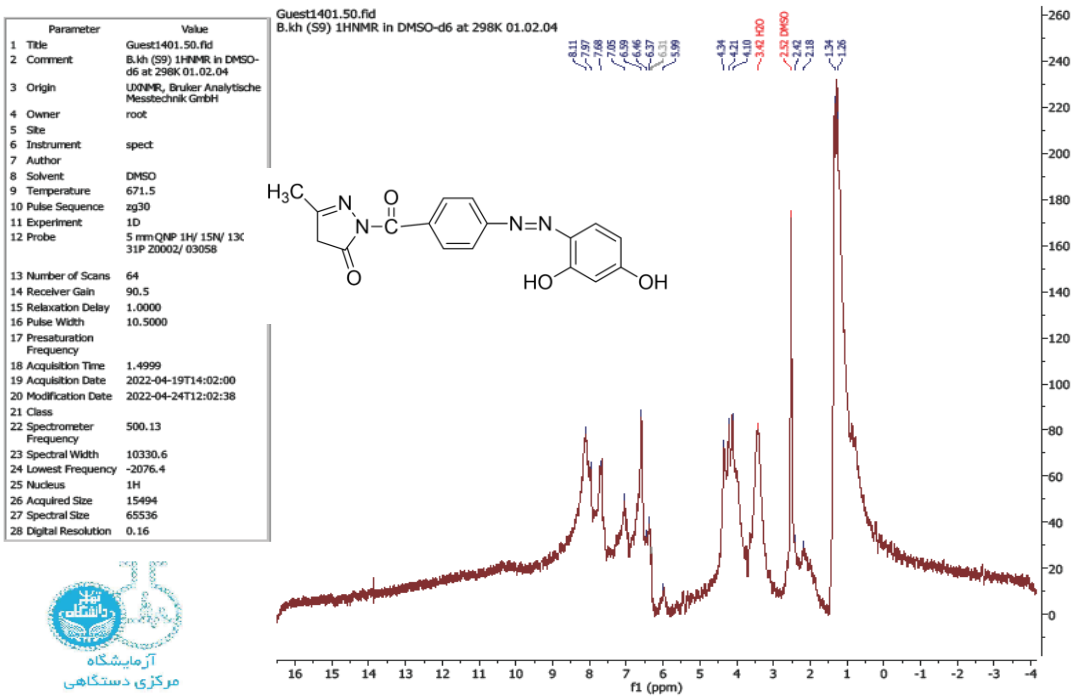


Figure3-26: ^{1}H-NMR spectrum of compound [S9]

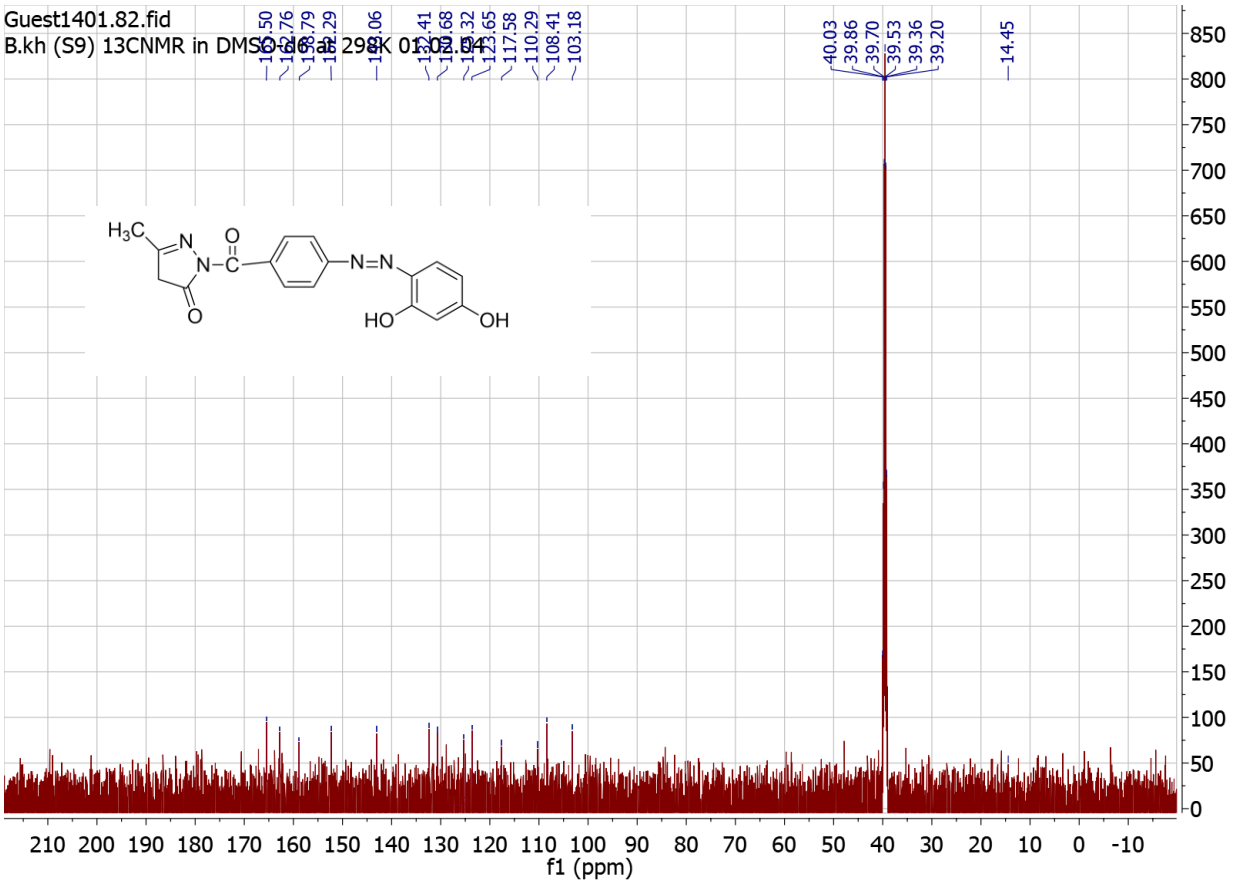
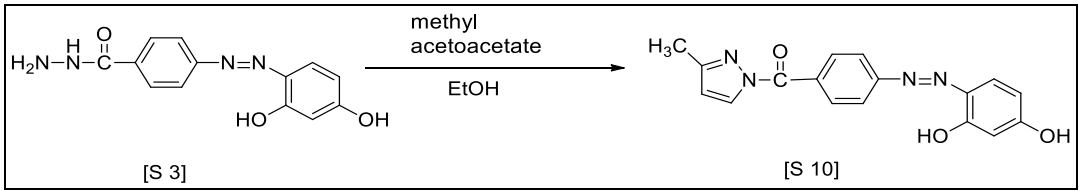


Figure3-27: ^{13}CNMR spectrum of compound [S9]

Compound [S10]:



Equation 3-10: Synthesis of Compound[S10]

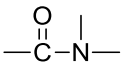
The FTIR spectra of compound [S10] show disappearance of NH_2 and NH bands in the region (3423,3346,3223) cm^{-1} and appearance of $C=O_{amide}$ band at (1701)cm^{-1}. and appearance of C-H aliphatic at (2978) cm^{-1} and C=C aromatic at (1518) cm^{-1}.

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) Spectrum of compound [S10] show appearance signal at 1.35 for (H,CH_3) and disappearance signal of (H,NH_2) and signal of (H,NH)

^{13}C-NMR (126 MHz,DMSO-d_6):(δ,ppm) spectrum show appearance signals at 108-148 for Ar-C and signal at 165 for $C=O_{amide}$

IR (ν,cm^{-1}): O-H (3367) ,C-H_{Ar} (3064) ,C-H $_{aliph}$(2978) ,C=O (1701) ,C=N (1602), $C=C_{Ar}$ (1518), N=N (1473).

^{1}H-NMR (δ, ppm) : (3H,CH_3): 1.35, (7H,Ar-H) :6.59 -8.10

^{13}C-NMR (δ, ppm) : 108 -148 for Ar-C , 165 for  amide

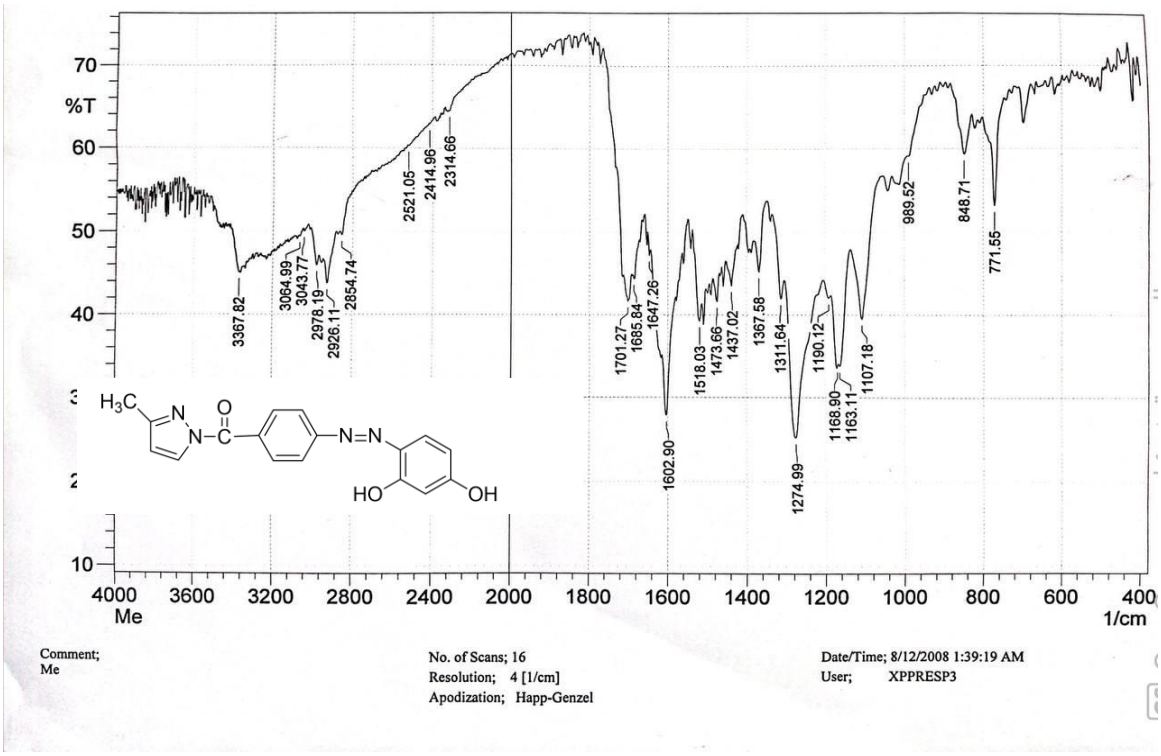


Figure3-28: F.T-IR spectrum of compound [S10]

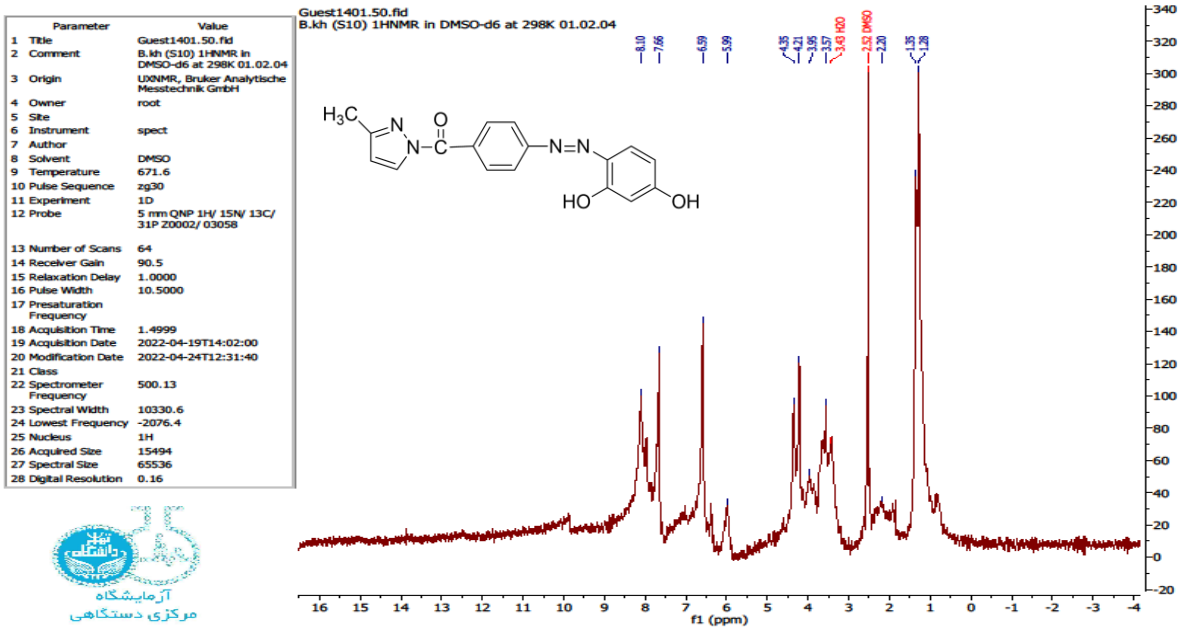


Figure 3-29 : ^{1}H-NMR spectrum of the compound [S10]

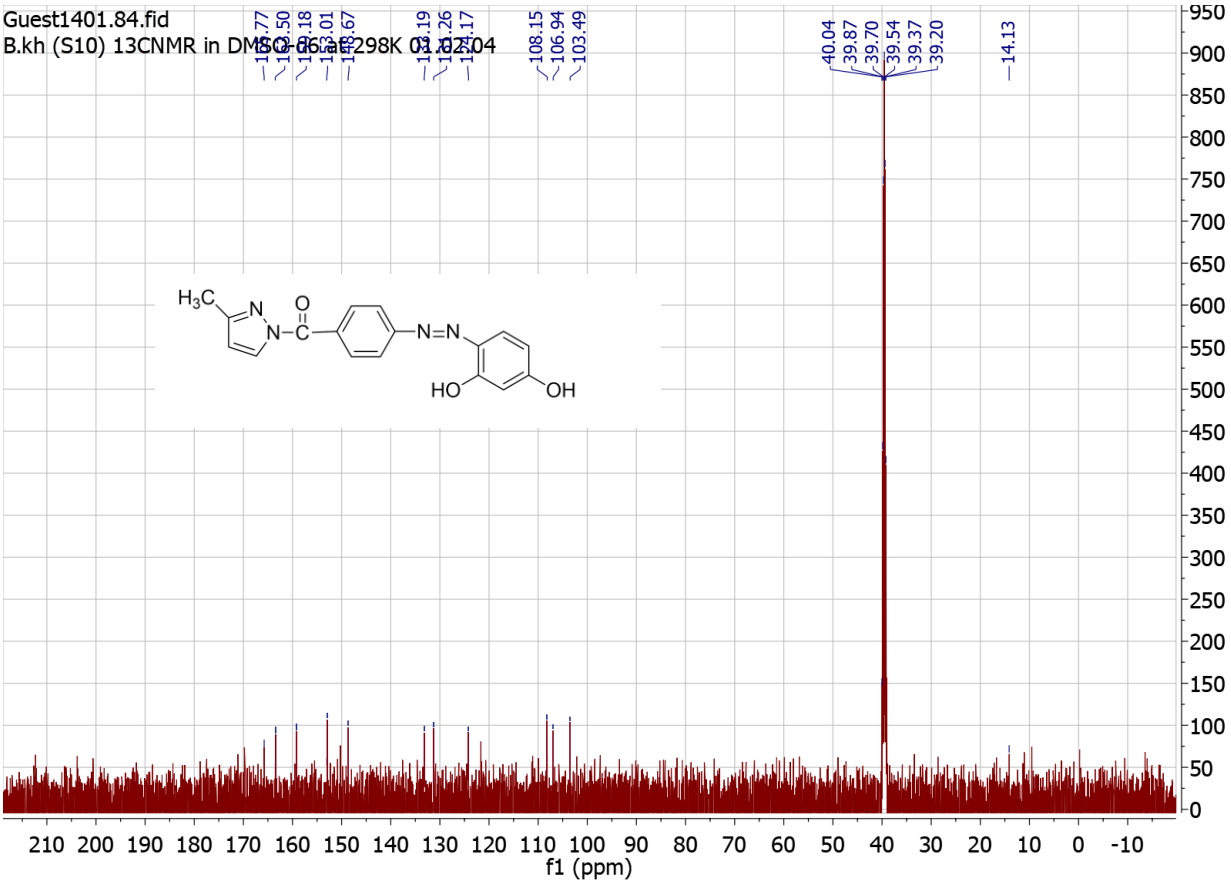
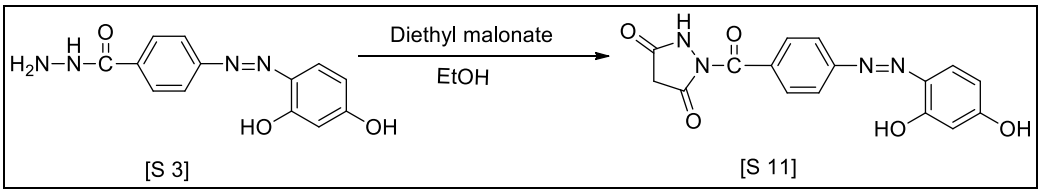


Figure3-30: ^{13}CNMR spectrum of compound [S10]

Compound [S11]:



Equation 3-11: Synthesis of Compound[S11]

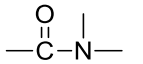
The FTIR spectra of compound [S11] show disappearance of NH_2 and NH bands in the region (3423,3346,3223) cm^{-1} and appearance of $C=O_{amide}$ band at (1714)cm^{-1}. and appearance of C=C aromatic at (1577) cm^{-1}.

^{1}H-NMR (500MHz,DMSO-d_6) :(δ, ppm) Spectrum of compound [S11] show appearance signal at 4.35 for (H,NH) and disappearance signal of (H,NH_2)

^{13}C-NMR (126 MHz,DMSO-d_6):(δ,ppm) spectrum show appearance signals at 118-132 for Ar-C and signal at 165 for $C=O_{amide}$

IR (ν,cm^{-1}) : O-H (3348) ,C-H_{Ar} (3061) ,C=O (1714) ,C=N (1602), ,C=C_{Ar} (1577) ,N=N (1518)

^{1}H-NMR (δ, ppm) : (H,NH) : 4.35, (7H,Ar-H) :7.62-8.12 .

^{13}C-NMR (δ, ppm) : 118-132 for Ar-C ,165 for  amide

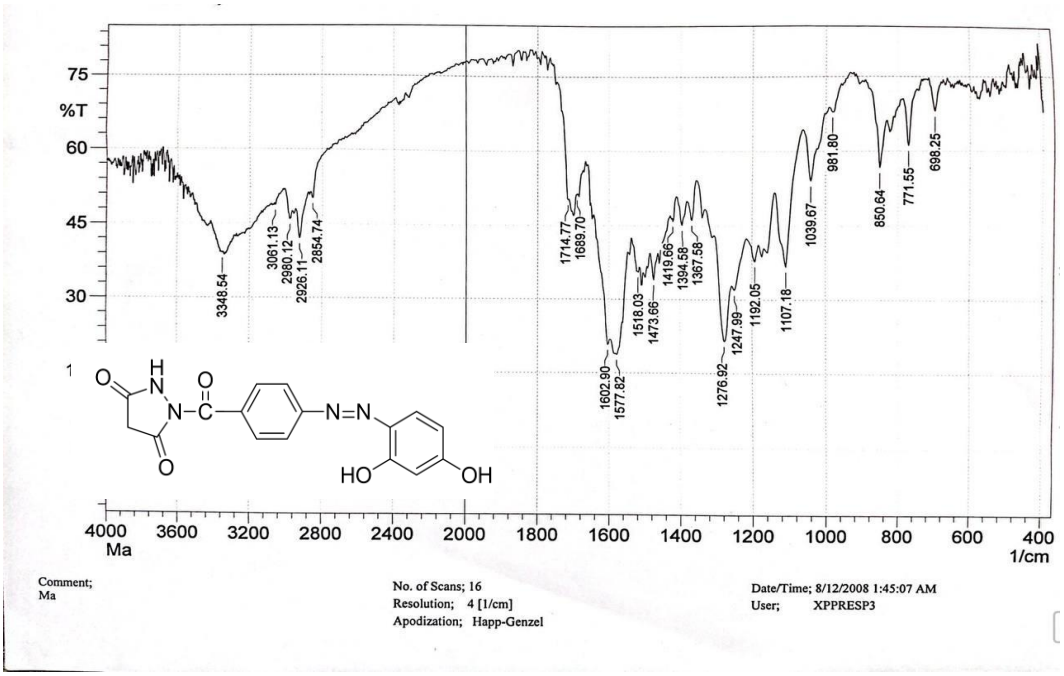
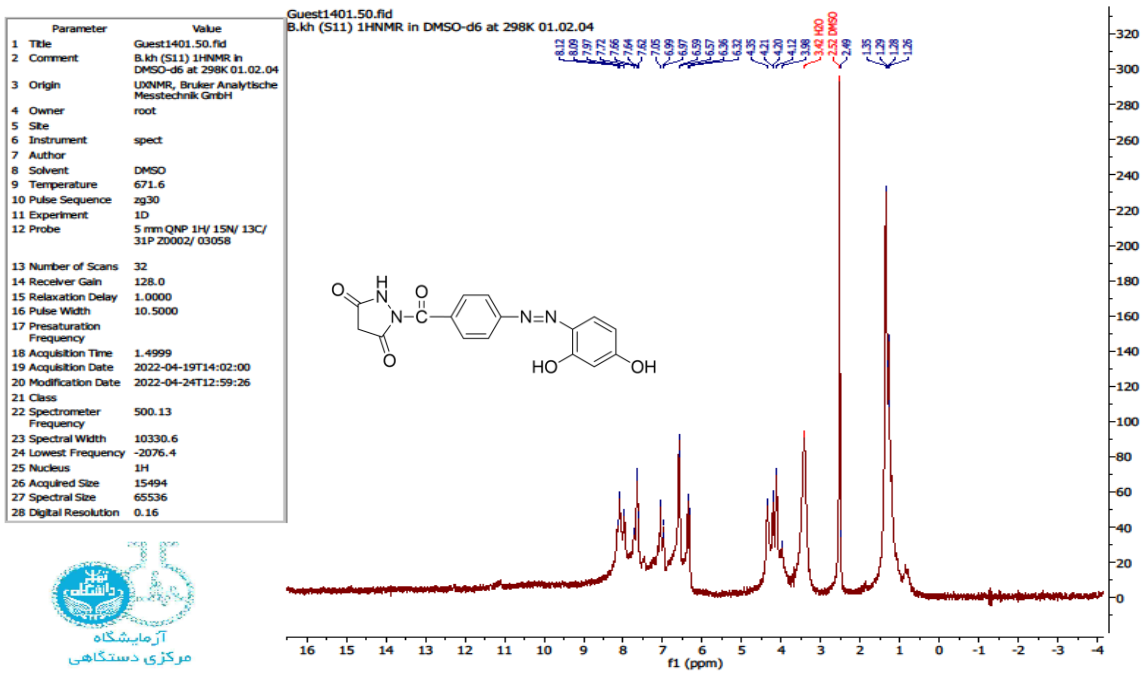


Figure3-31: F.T-IR spectrum of compound [S11]



	Parameter	Value
1	Title	Guest1401.50.fid
2	Comment	B.kh (S11) 1HNMR in DMSO-d6 at 298K 01.02.04
3	Origin	UXNMR, Bruker Analytische Messtechnik GmbH
4	Owner	root
5	Site	
6	Instrument	spect
7	Author	
8	Solvent	DMSO
9	Temperature	671.6
10	Pulse Sequence	zg30
11	Experiment	1D
12	Probe	5 mm QNP 1H/ 15N/ 13C/ 31P Z0002/ 03058
13	Number of Scans	32
14	Receiver Gain	128.0
15	Relaxation Delay	1.0000
16	Pulse Width	10.5000
17	Presaturation Frequency	
18	Acquisition Time	1.4999
19	Acquisition Date	2022-04-19T14:02:00
20	Modification Date	2022-04-24T12:59:26
21	Class	
22	Spectrometer Frequency	500.13
23	Spectral Width	10330.6
24	Lowest Frequency	-2076.4
25	Nucleus	1H
26	Acquired Size	15494
27	Spectral Size	65536
28	Digital Resolution	0.16

Figure3-32: ^{1}H-NMR spectrum of compound [S11]

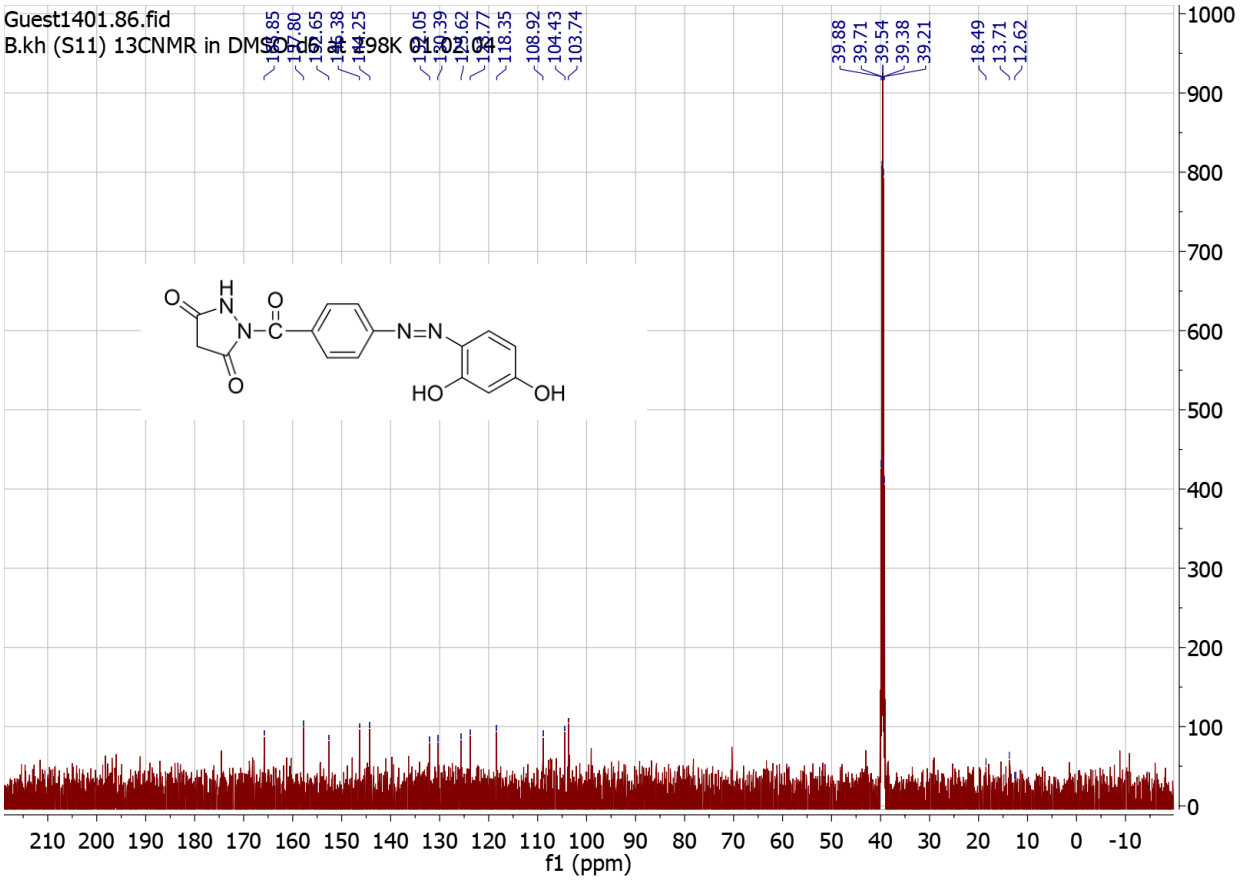
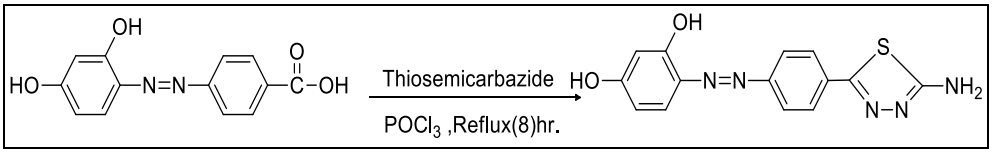


Figure3-33: ^{13}CNMR spectrum of compound [S11]

3.6 Synthesis of compound [S12]:

Cyclization of compound [S1] with thiosemicarbazide in presence phosphorous oxy chloride result compound[S12].



Equation 3-12 : Synthesis of Compound[S12]

The FTIR spectrum exhibit appearance band absorption of NH_2 at (3221 ,3327) cm^{-1} and appearance absorption band of C=N at (1608) cm^{-1} and disappearance band of C=O $_{carboxylic\ acid}$ at (1683) cm^{-1}.

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) Spectrum show disappearance signal of (H, COOH) and appearance signal of (H, NH_2) at 6.79 .

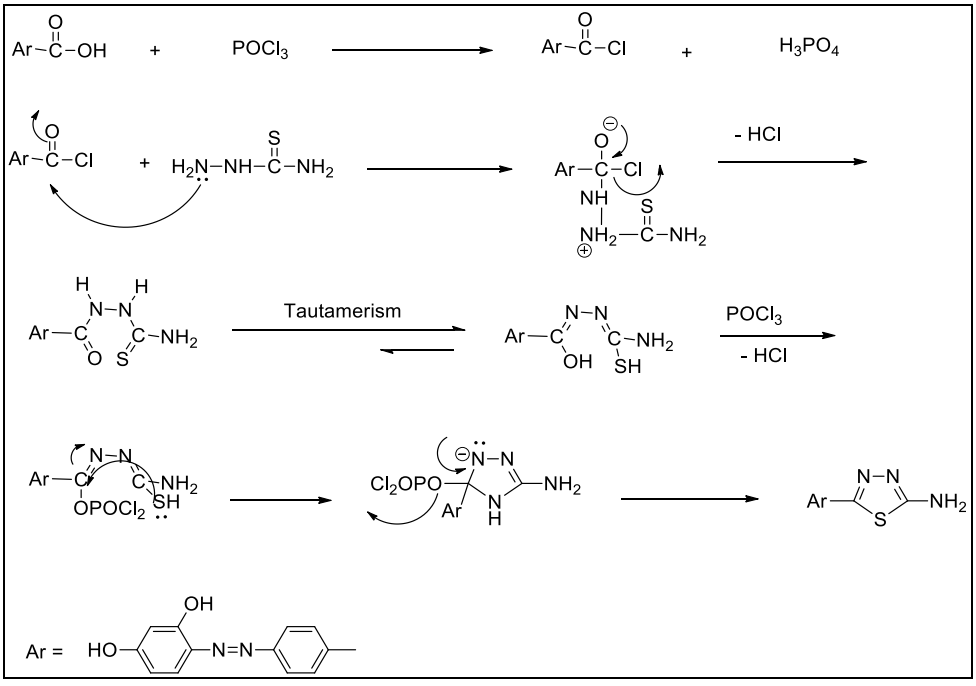
^{13}C-NMR (126 MHz,DMSO-d_6):(δ,ppm) spectrum show appearance signals at 117-135 for Ar-C and signal at 174 for C-S

IR (ν,cm^{-1}): O-H (3406) ,NH_2 (3327,3221) ,C-H_{Ar} (3122) ,C=N (1608) ,C=C_{Ar} (1577) ,N=N (1508) .

^{1}H-NMR (δ, ppm): (H,NH_2) 6.79 ,(7H,Ar-H) 6.81-8.19 .

^{13}C-NMR (δ, ppm) : (117-135) for Ar-C ,174 for C-S

The mechanism of synthesis thiadiazole is shown below[(94)]:



Scheme 3-5:Mechanism of synthesis compound [S12]

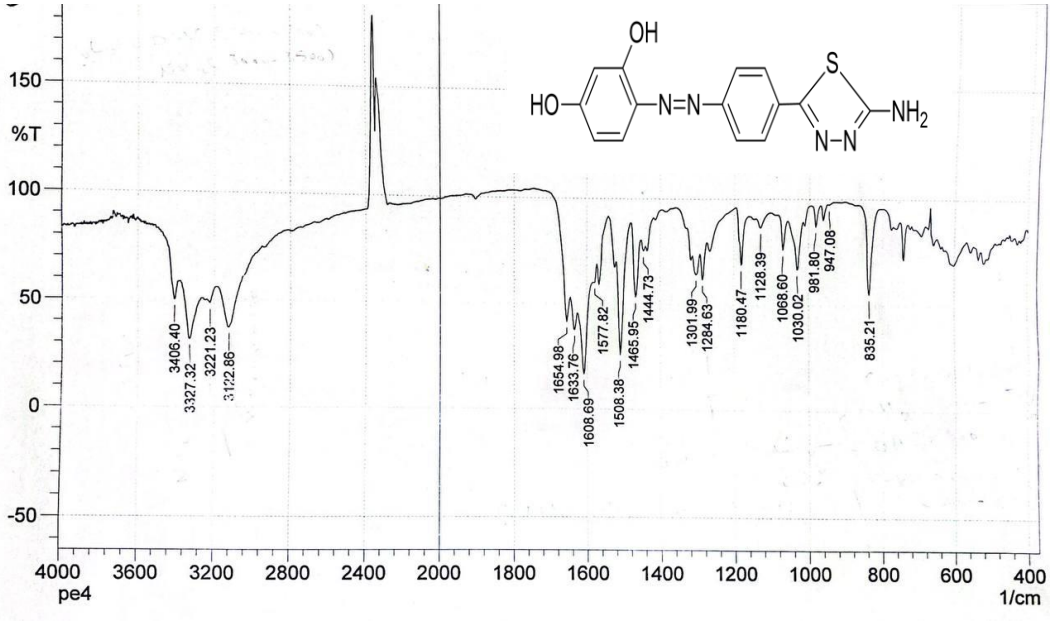


Figure3-34: FT.IR spectrum of compound [S12]

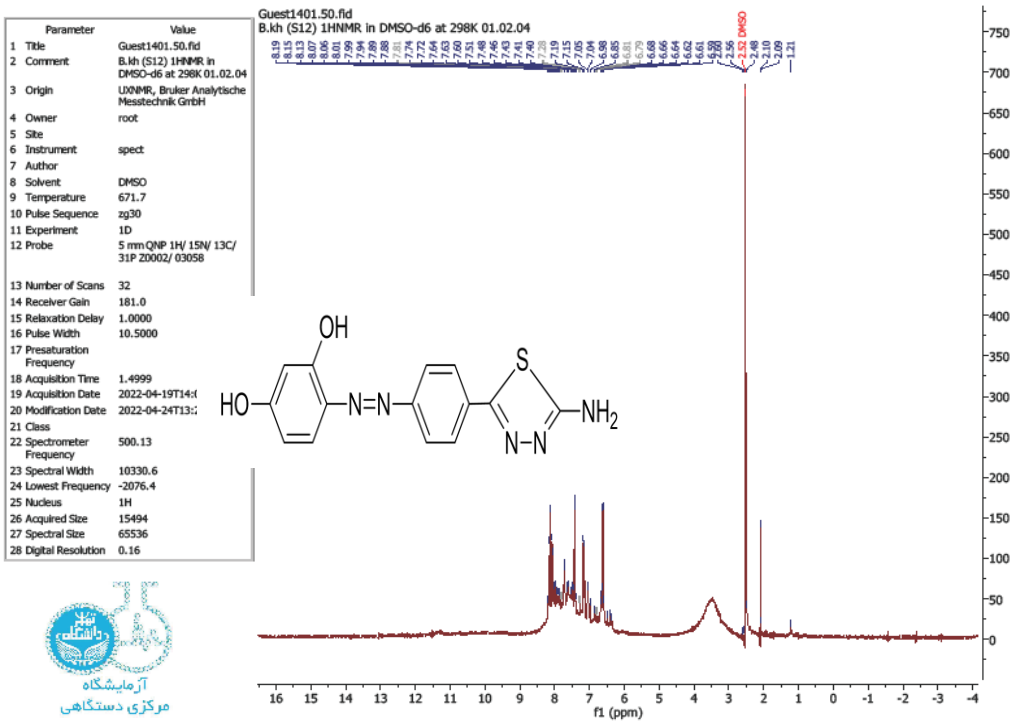


Figure3-35: ^{1}H-NMR spectrum of compound [S12]

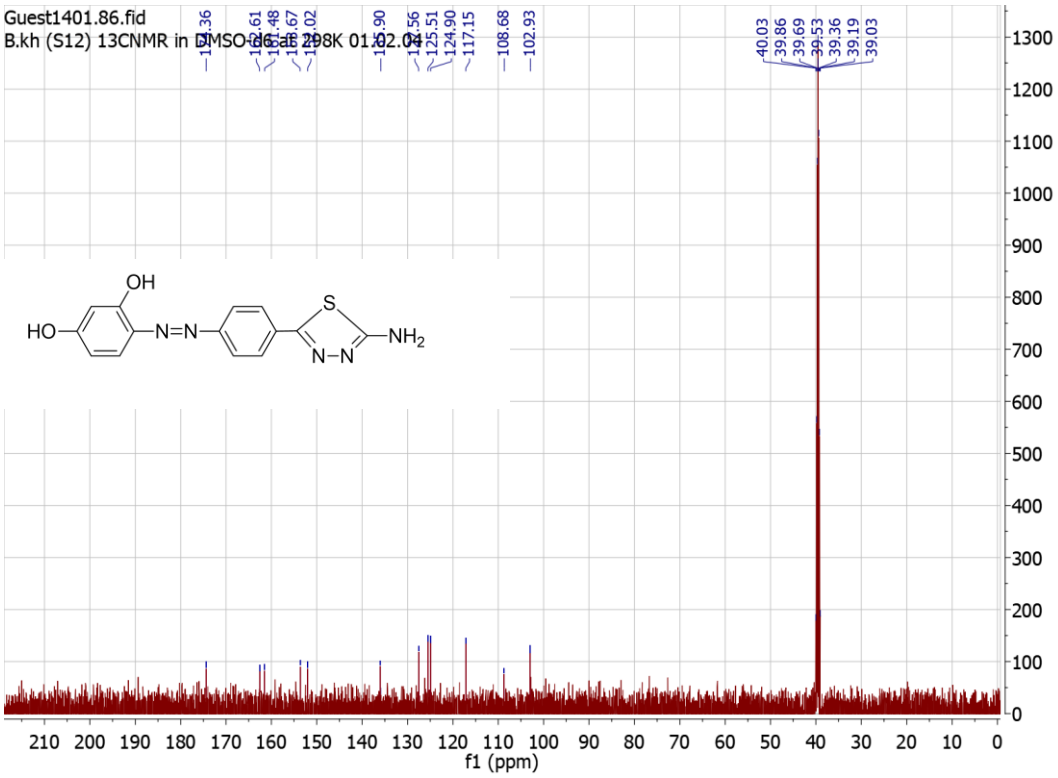


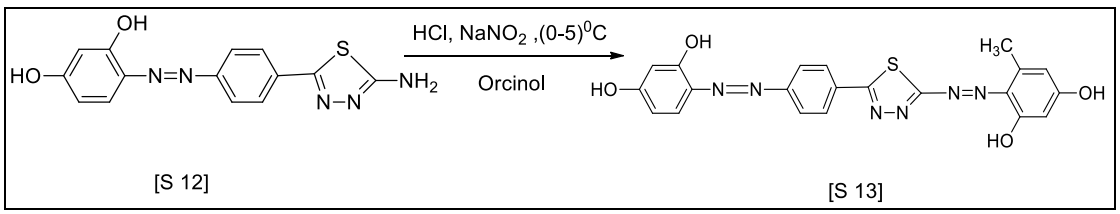
Figure3-36: ^{13}CNMR spectrum of compound [S12]

3.7 Synthesis of azo compounds from thiadiazole [S13-S23]:

The azo compounds [S13-S23] were synthesis by reaction of two organic compounds ,a diazonium salt (1,3,4-thiadiazole amine) reacts as an electrophile with a coupling molecule that is rich in electrons. (orcinol ,hydroquinone ,resorcinol, catechol, alpha naphthol ,beta naphthol,2-methyl naphthol,2,6-dimethyl phenol,1,2,4-triazole,thiol 1,2,4-triazole ,

p-touldine) respectively at (0-5 ^{0}C).

Compound [S13]:



Equation 3-13: Synthesis of Compound [S13]

The FTIR spectrum of compound [S13] exhibited absorption band at (1473) cm^{-1} for N=N and disappearance absorption band at (3327,3221) cm^{-1} for NH_2

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) spectrum show appearance signal at 6.36-8.07 for (H,Ar-H) and signal at 1.12 for (H,CH_3) and disappearance signal of (H,NH_2) .

^{13}C-NMR (126 MHz,DMSO-d_6) :(δ, ppm) spectrum show signal at 20 for CH_3 and signals at 117-140 for Ar-C and signal at 179 for C-S

IR (*v*, cm^{-1}): O-H (3387),C-H $_{Ar}$ (3066), C=N (1602), C=C_{Ar} (1541), N=N (1473).

^{1}H-NMR (δ, ppm): (3H,CH_3):1.12, (9H,Ar-H) : 6.36 -8.07 .

^{13}C-NMR (δ, ppm) : 20 for CH_3 , (117-140) for Ar-C ,179 for C-S

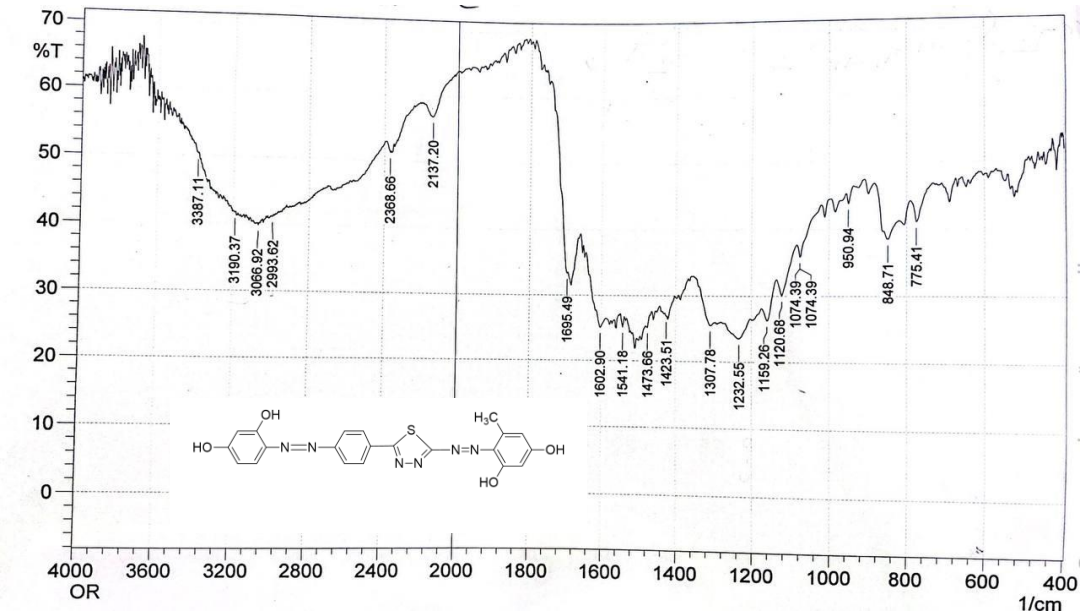
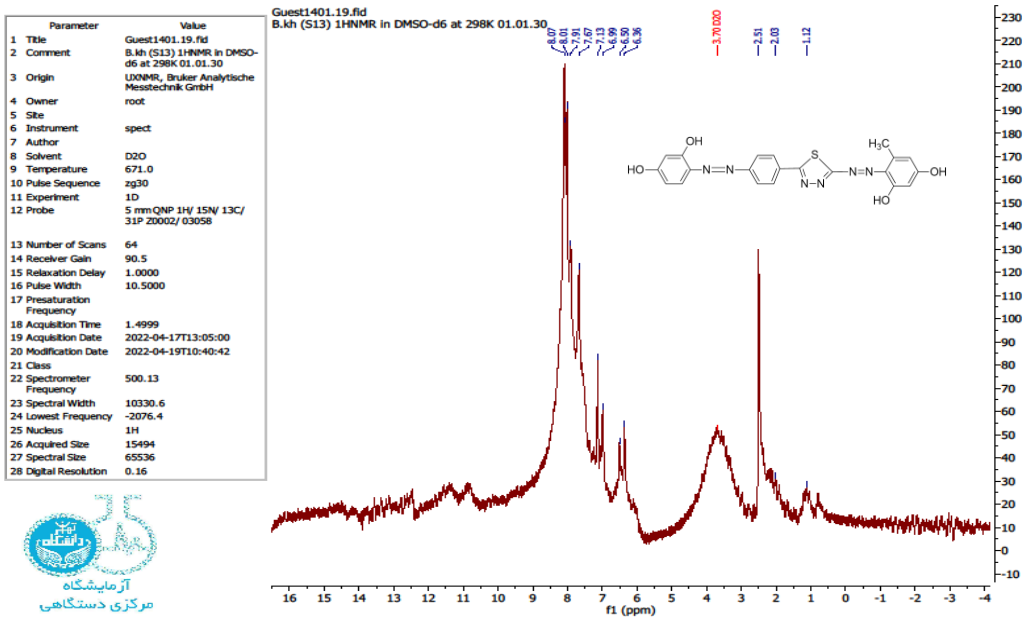


Figure3-37: F.T-IR spectrum of compound [S13]



Parameter	Value
1 Title	Guest1401.19.fid
2 Comment	B.kh (S13) 1HNMR in DMSO-d6 at 298K 01.01.30
3 Origin	UXNMR, Bruker Analytische Messtechnik GmbH
4 Owner	root
5 Site	
6 Instrument	spect
7 Author	
8 Solvent	D2O
9 Temperature	671.0
10 Pulse Sequence	zg30
11 Experiment	1D
12 Probe	5 mm QNP 1H/ 15N/ 13C/ 31P Z0002/ 03058
13 Number of Scans	64
14 Receiver Gain	90.5
15 Relaxation Delay	1.0000
16 Pulse Width	10.5000
17 Presaturation Frequency	
18 Acquisition Time	1.4999
19 Acquisition Date	2022-04-17T13:05:00
20 Modification Date	2022-04-19T10:40:42
21 Class	
22 Spectrometer Frequency	500.13
23 Spectral Width	10330.6
24 Lowest Frequency	-2076.4
25 Nucleus	1H
26 Acquired Size	15494
27 Spectral Size	65536
28 Digital Resolution	0.16

Figure3-38: ^{1}H-NMR spectrum of compound [S13]

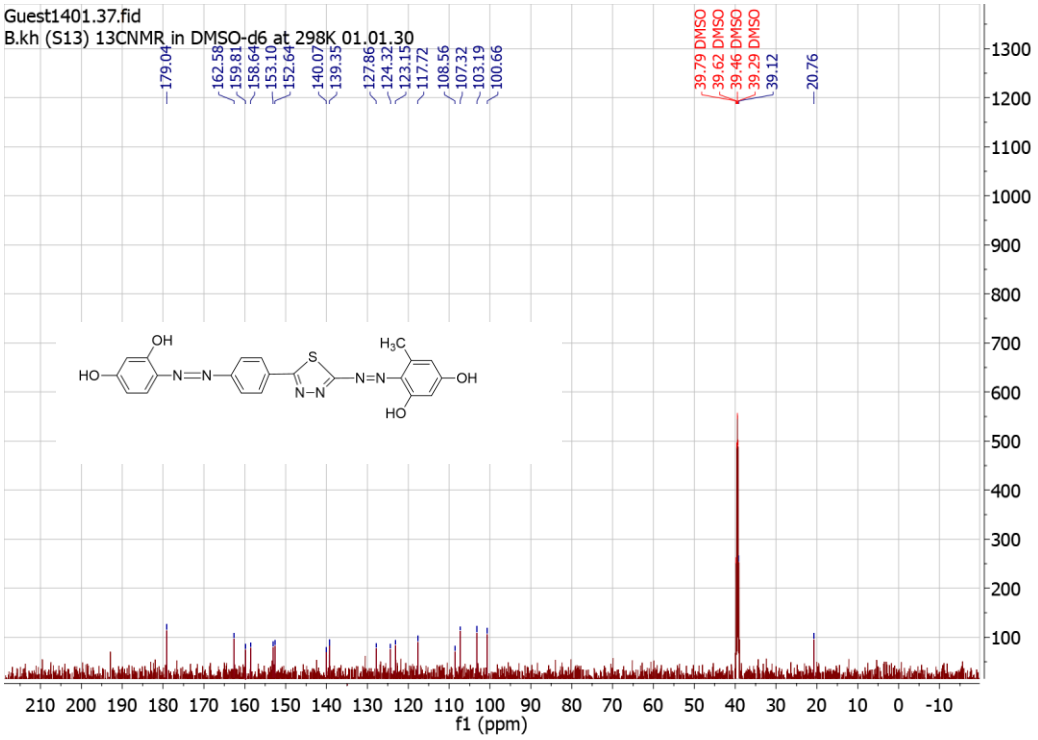
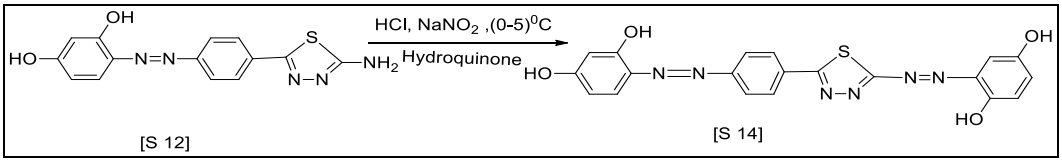


Figure3-39: ^{13}CNMR spectrum of compound [S13]

Compound [S14]:



Equation 3-14: Synthesis of Compound [S14]

The FTIR spectrum of compound [S14] exhibited absorption band at (1491) cm^{-1} for N=N and disappearance absorption band at (3327,3221) cm^{-1} for NH_2

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) spectrum show appearance signal at 7.03 - 8.13 for (H,Ar-H) and disappearance a signal of (H,NH_2)

IR (*v*,cm^{-1}): O-H (3489,3335), ,C-H $_{Ar}$ (3080),C=N (1600) , C=C_{Ar} (1568), N=N (1491).

^{1}H-NMR (δ, ppm): (10 H,Ar-H) : 7.03 -8.13,(H,OH):13.9

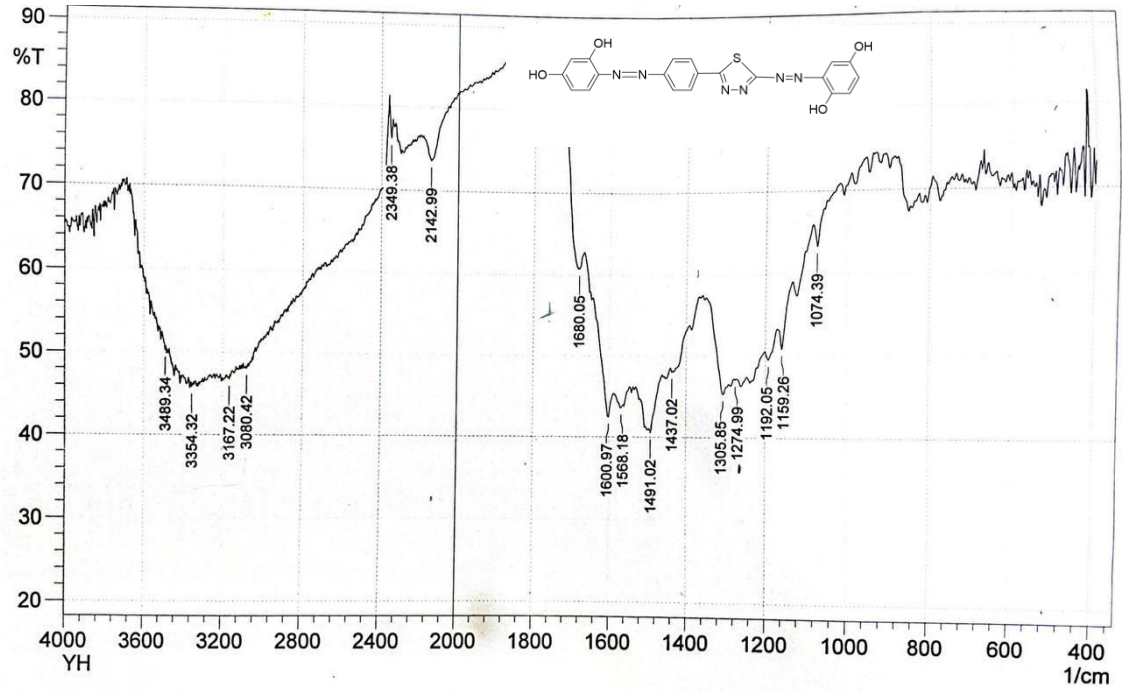


Figure3-40: F.T-IR spectrum of compound [S14]

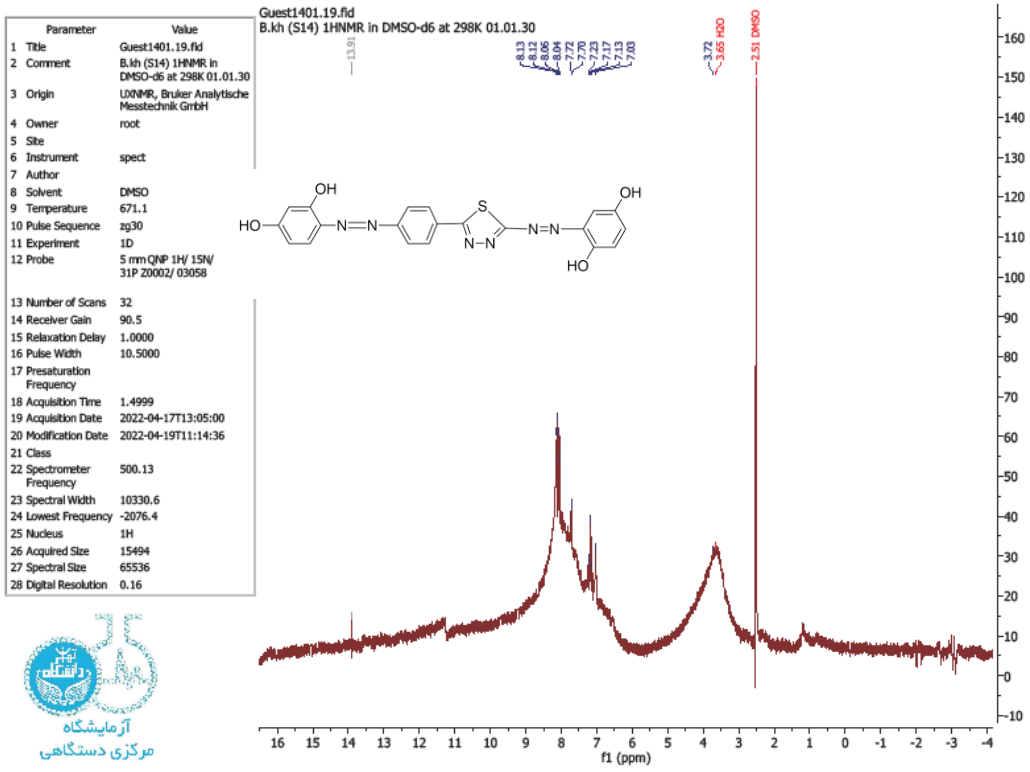
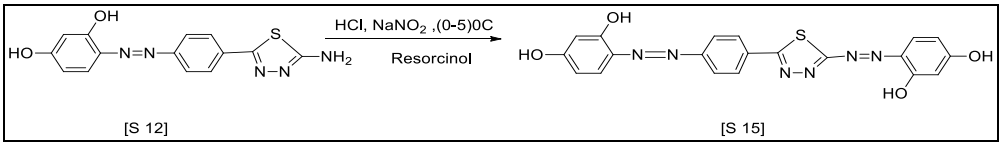


Figure3-4: ^{1}H-NMR spectrum of compound [S14]

Compound [S15] :



Equation 3-15: Synthesis of Compound [S15]

The FTIR spectrum of compound [S15] exhibited absorption band at (1506) cm^{-1} for N=N and disappearance absorption band at (3327,3221) cm^{-1} for NH_2

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) Spectrum show appearance signal at 6.2 - 8.15 for (H,Ar-H) and disappearance a signal of (H,NH_2)

^{13}C-NMR (126 MHz,DMSO-d_6) :(δ, ppm) spectrum show signals at 116-131 for Ar-C and signal at 174 for C-S

IR (*v*, cm^{-1}): O-H (3441,3277),C-H $_{Ar}$ (3078) ,C=N (1602) , C=C_{Ar} (1506) , N=N (1506).

^{1}H-NMR (δ, ppm): (10H,Ar-H) : 6.2 -8.15

^{13}C-NMR (δ, ppm) : (116-131) for Ar-C ,174 for C-S

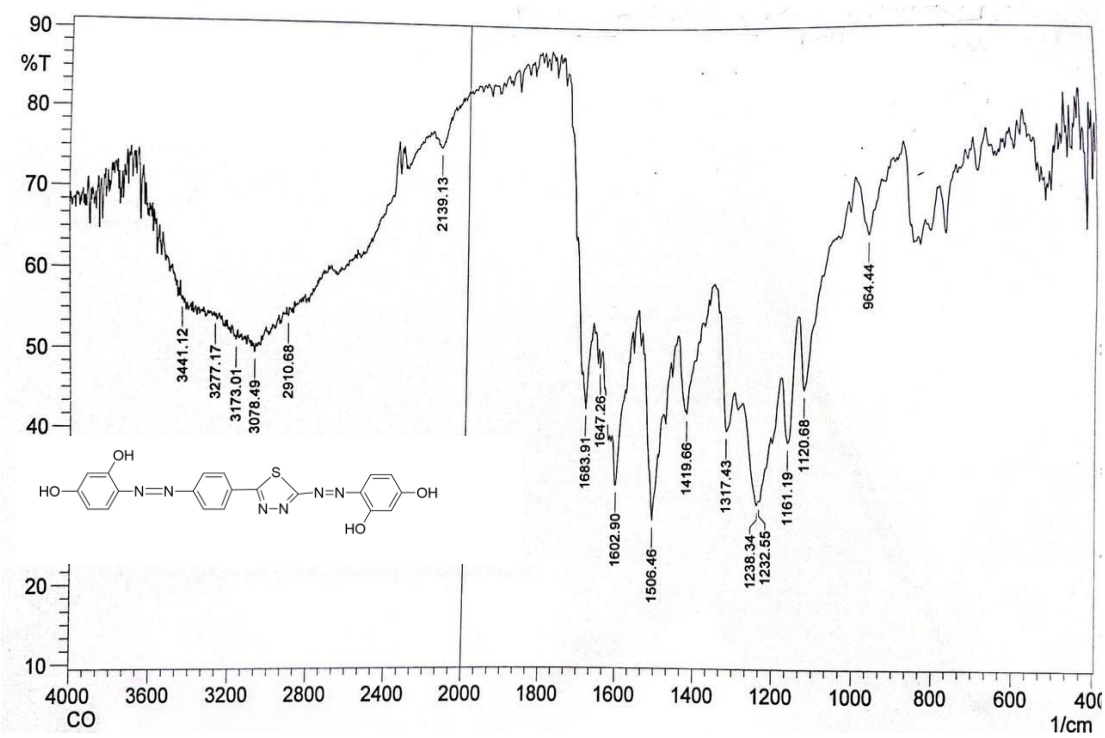


Figure3-42: F.T-IR spectrum of compound [S15]

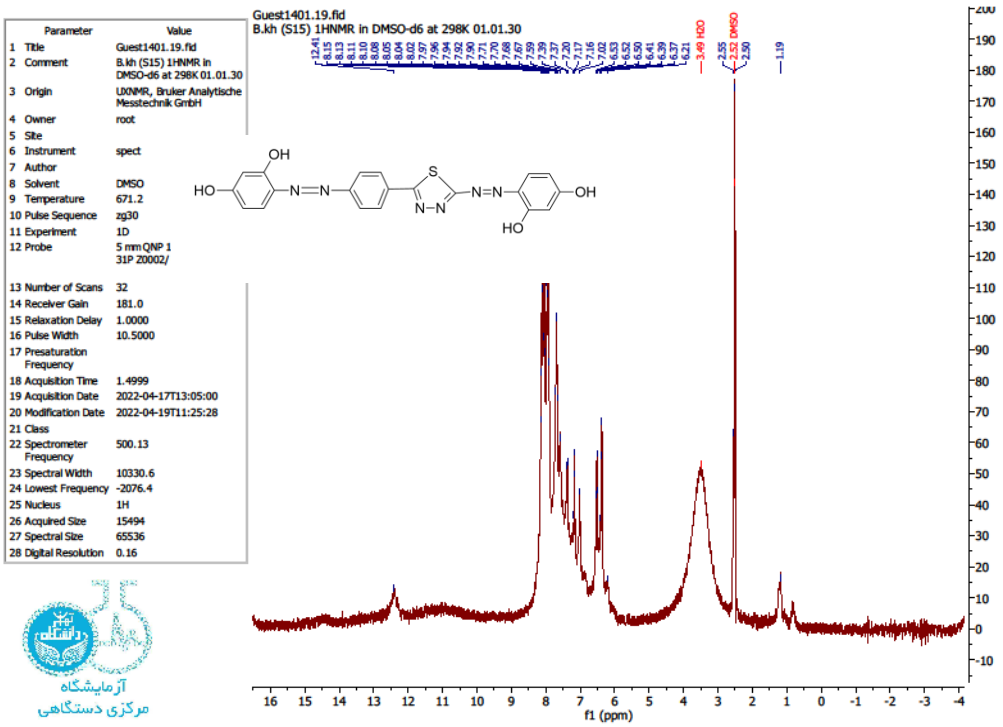


Figure3-43: ^{1}H-NMR spectrum of compound [S15]

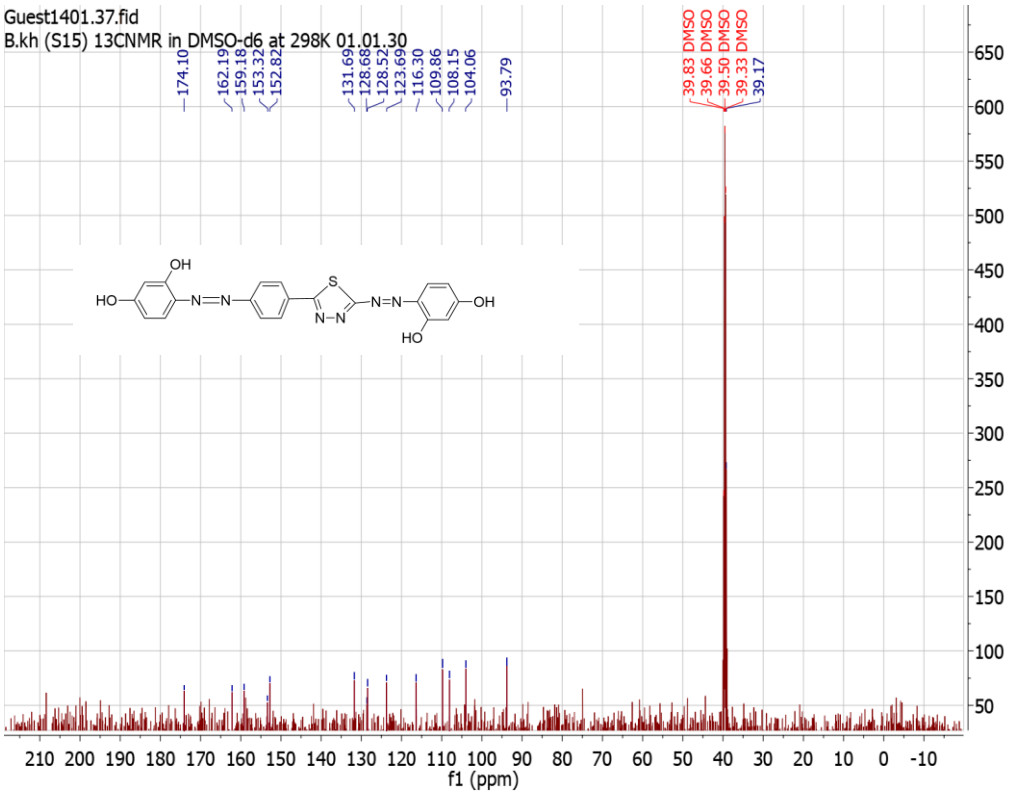
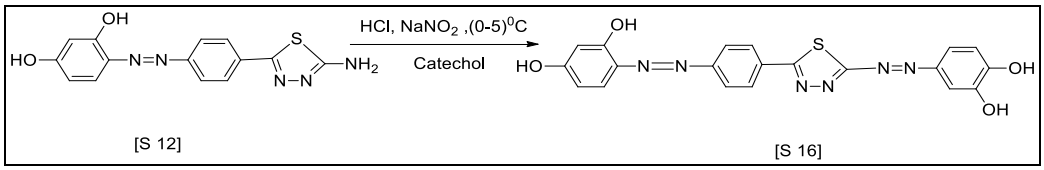


Figure3-44: ^{13}CNMR spectrum of compound [S15]

Compound [S16]:



Equation 3-16: Synthesis of Compound [S16]

The FTIR spectrum of compound [S16] exhibited absorption band at (1521) cm^{-1} for N=N and disappearance absorption band at (3327,3221) cm^{-1} for NH_2

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) spectrum show appearance signal at 6.36-8.14 for (H,Ar-H) and disappearance signal of (H,NH_2) .

^{13}C-NMR (126 MHz,DMSO-d_6) :(δ, ppm) spectrum show signals at 117-162 for Ar-C and signal at 174 for C=S

IR (ν, cm^{-1}): O-H (3443,3273) , C-H $_{Ar}$ (3020),C=N (1602) ,C=C_{Ar} (1560) , N=N (1521).

^{1}H-NMR (δ, ppm): (10 H,Ar-H) : 6.36 -8.14 .

^{13}C-NMR (δ, ppm) : (117-162) for Ar-C ,174 for C=S

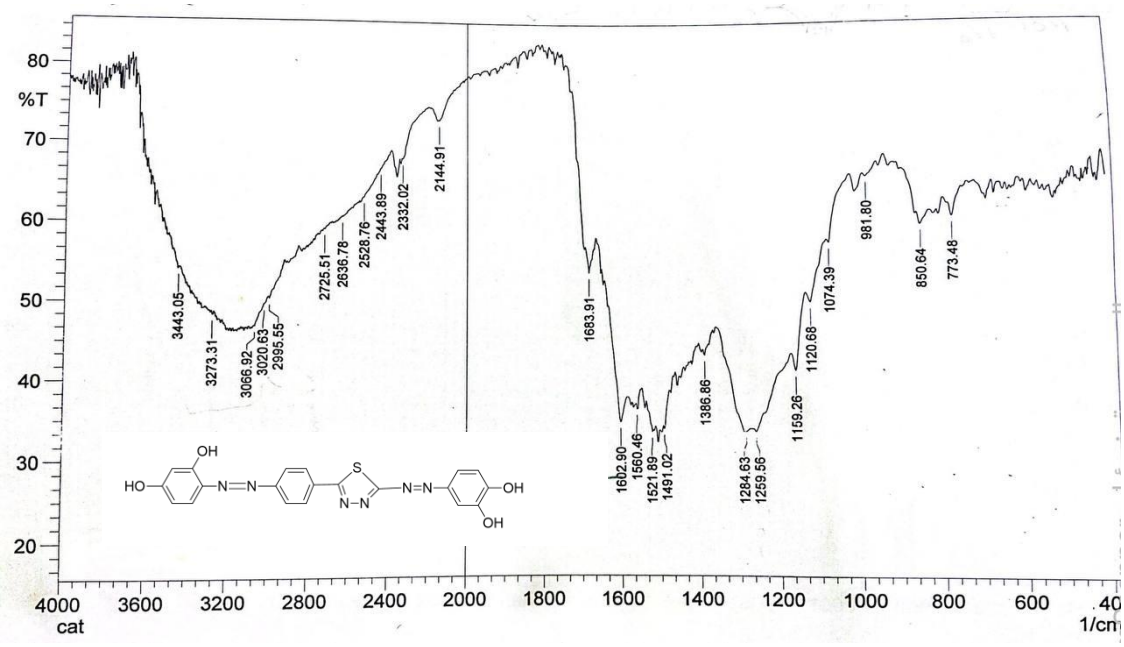


Figure3-45: F.T-IR spectrum of compound [S16]

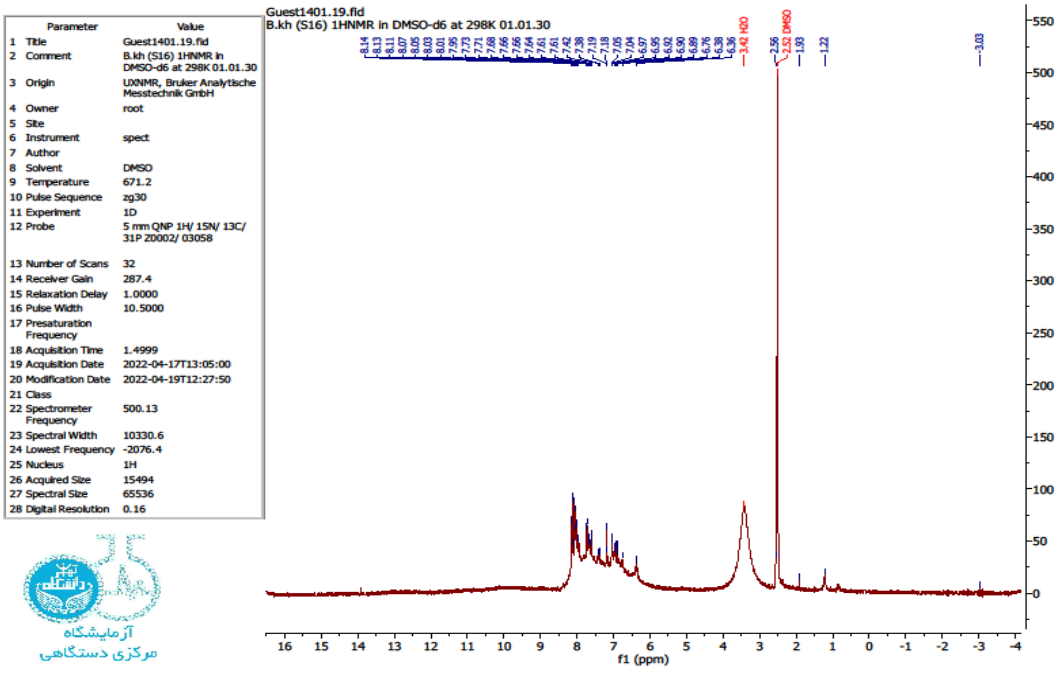


Figure3-46: ^{1}H-NMR spectrum of compound [S16]

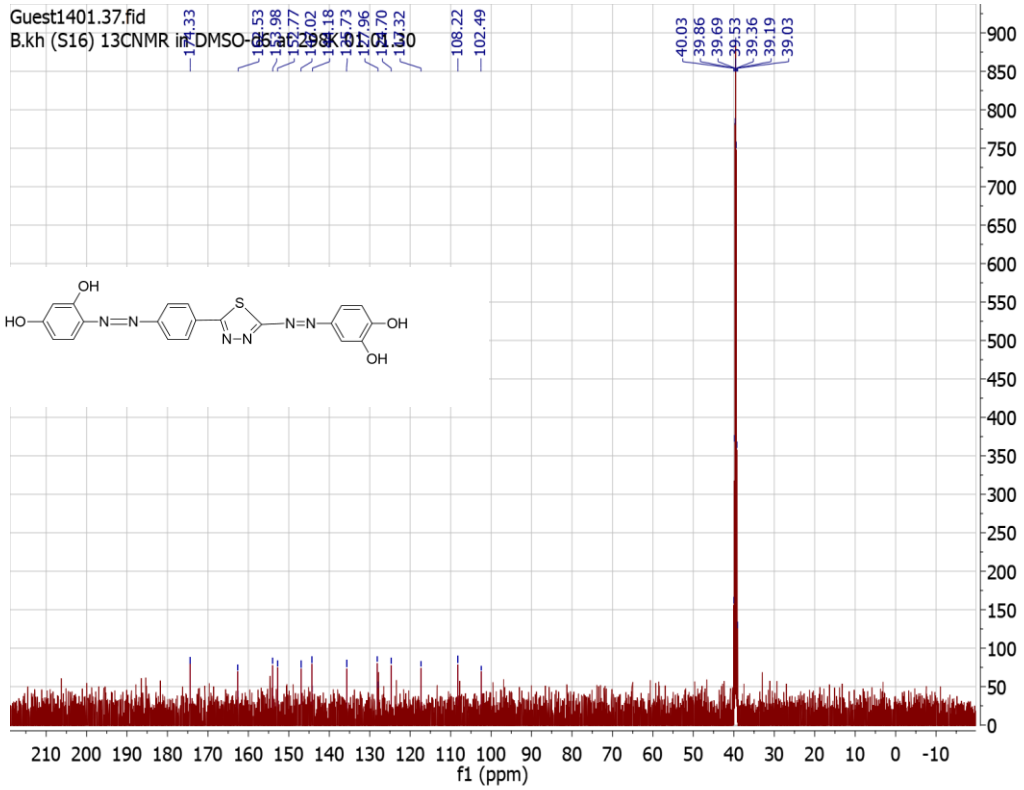
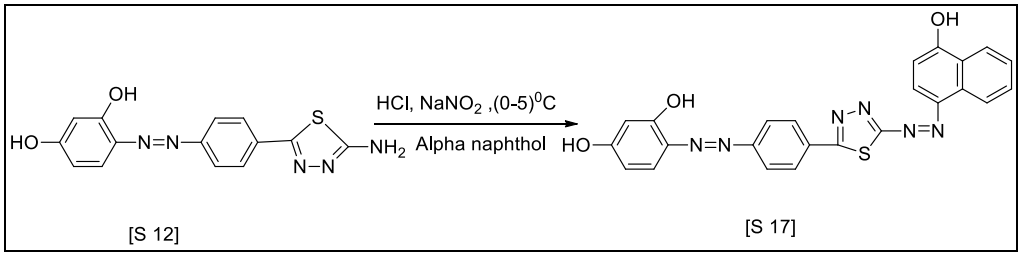


Figure3-47: ^{13}CNMR spectrum of compound [S16]

Compound [S17]:



Equation 3-17: Synthesis of Compound [S17]

The FTIR spectrum of compound [S17] exhibited absorption band at (1419) cm^{-1} for N=N and disappearance absorption band at (3327,3221) cm^{-1} for NH_2

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) spectrum show appearance signal at 6.38-8.13 for (H,Ar-H) and disappearance signal of (H,NH_2) .

IR (ν, cm^{-1}): O-H (3373,327) ,C-H $_{Ar}$ (3099), C=N (1602), C=C_{Ar} (1502),N=N (1419).

^{1}H-NMR (δ, ppm): (13H,Ar-H) :6.38 -8.13 .

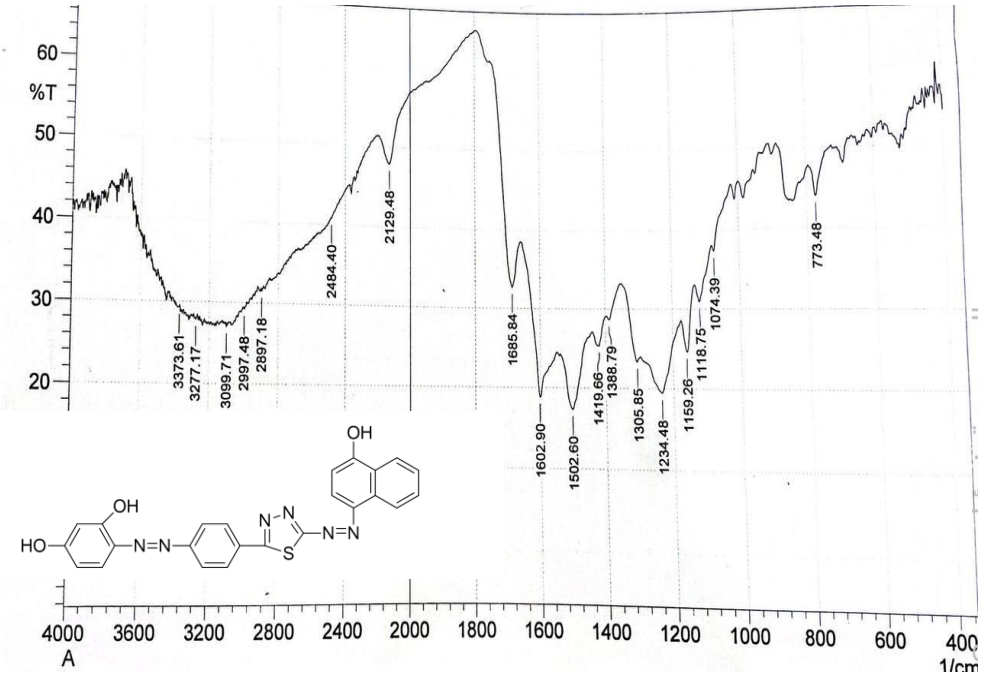


Figure3-48: F.T-IR spectrum of compound [S17]

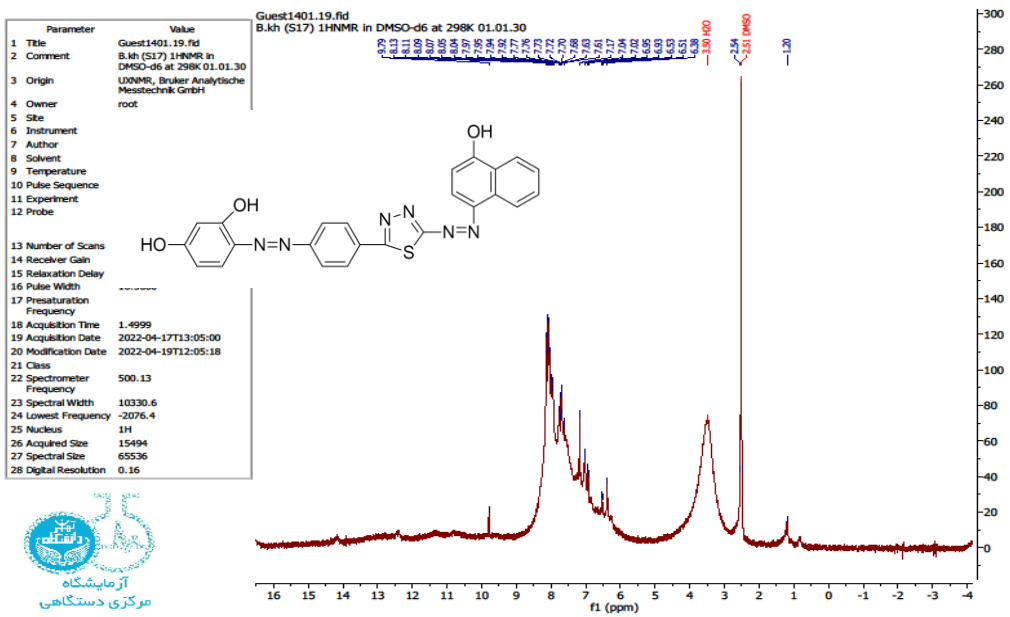
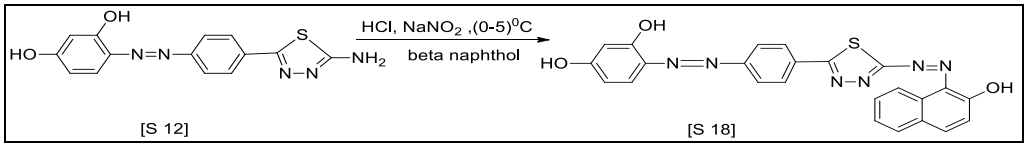


Figure3-49: ^{1}H-NMR spectrum of compound [S17]

Compound [S18]:



Equation 3-18: Synthesis of Compound [S18]

The FTIR spectrum of compound [S18] exhibited absorption band at (1498) cm^{-1} for N=N and disappearance absorption band at (3327,3221) cm^{-1} for NH_2

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) spectrum show appearance signal at 6.42-8.18 for (H,Ar-H) and disappearance signal of (H,NH_2) .

^{13}C-NMR (126 MHz,DMSO-d_6) :(δ, ppm) spectrum show signals at 123-162 for Ar-C and signal at 172 for C-S

IR (ν, cm^{-1}): O-H (3213), ,C-H $_{Ar}$ (2951),C=N (1600) , C=C_{Ar} (1560) , N=N (1498).

^{1}H-NMR (δ, ppm): (13 H,Ar-H) : 6.42 - 8.18

^{13}C-NMR (δ, ppm) : (123-162) for Ar-C ,172 for C-S

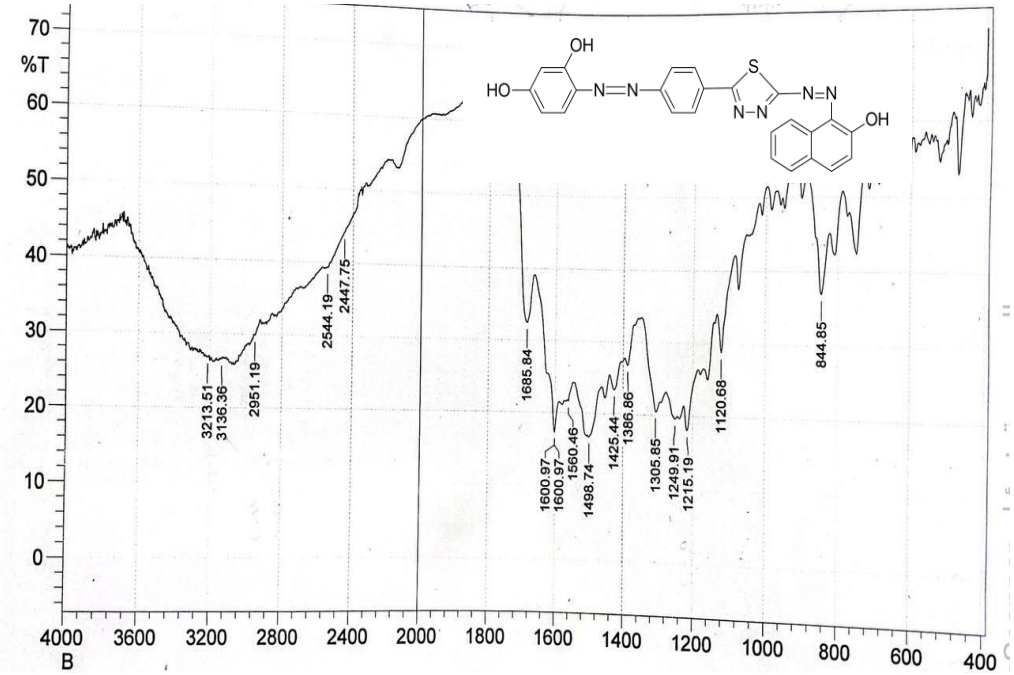


Figure3-50: F.T-IR spectrum of compound [S18]

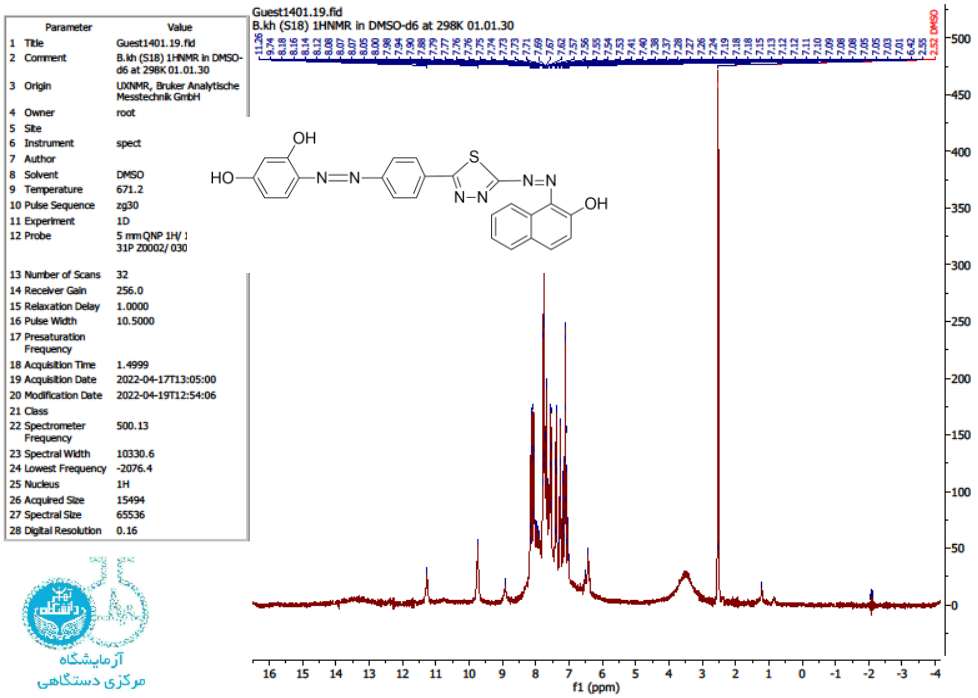


Figure 3-51: ^{1}H-NMR spectrum of compound [S18]

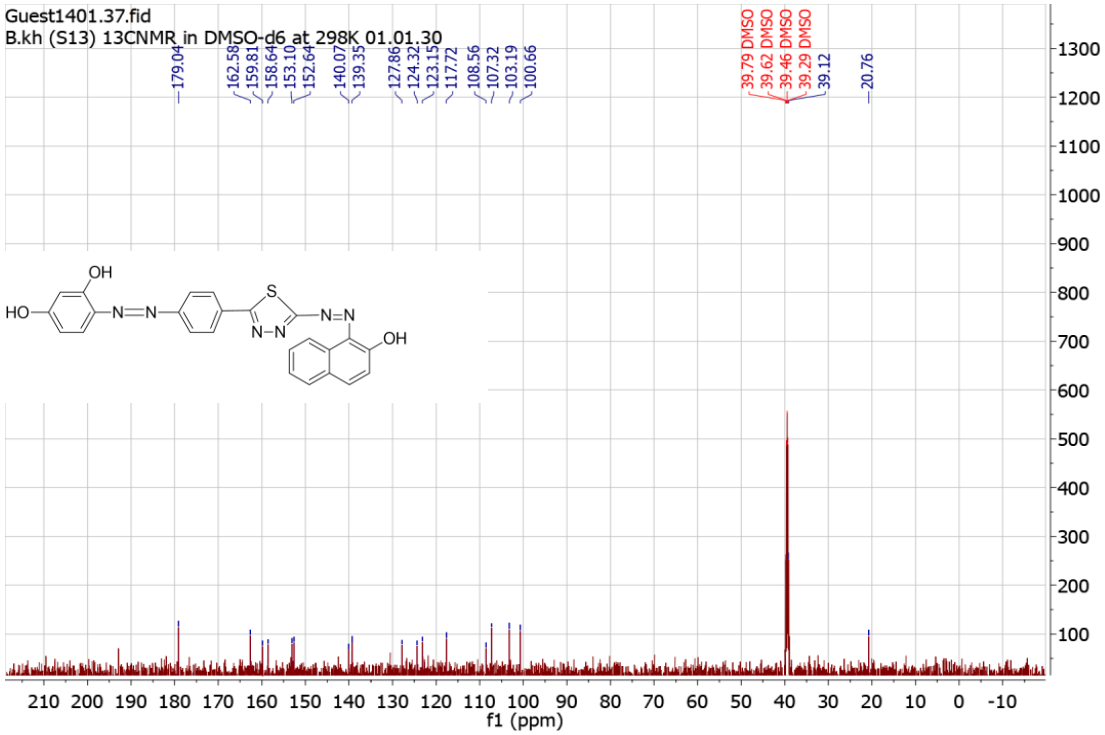
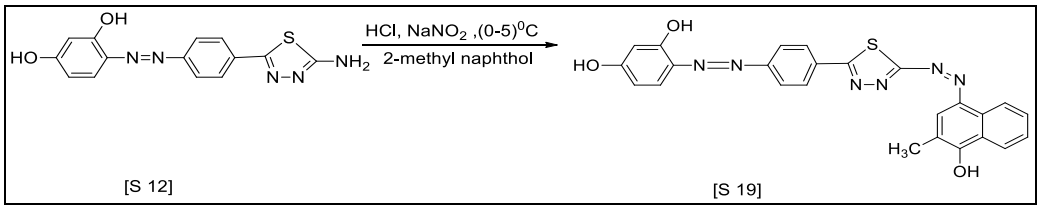


Figure3-52: ^{13}CNMR spectrum of compound [S18]

Compound [S19]:



Equation 3-19: Synthesis of Compound [S19]

The FTIR spectrum of compound [S19] exhibited absorption band at (1595) cm^{-1} for N=N and disappearance absorption band at (3327,3221) cm^{-1} for NH_2

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) spectrum show appearance signal at 6.87-8.21 for (H,Ar-H) and disappearance signal of (H,NH_2) .

IR (ν, cm^{-1}): O-H (3377) ,C-H $_{Ar}$ (3045), C=N (1683) , C=C_{Ar} (1635) N=N (1595).

^{1}H-NMR (δ, ppm): (12H,Ar-H) :6.87-8.21.

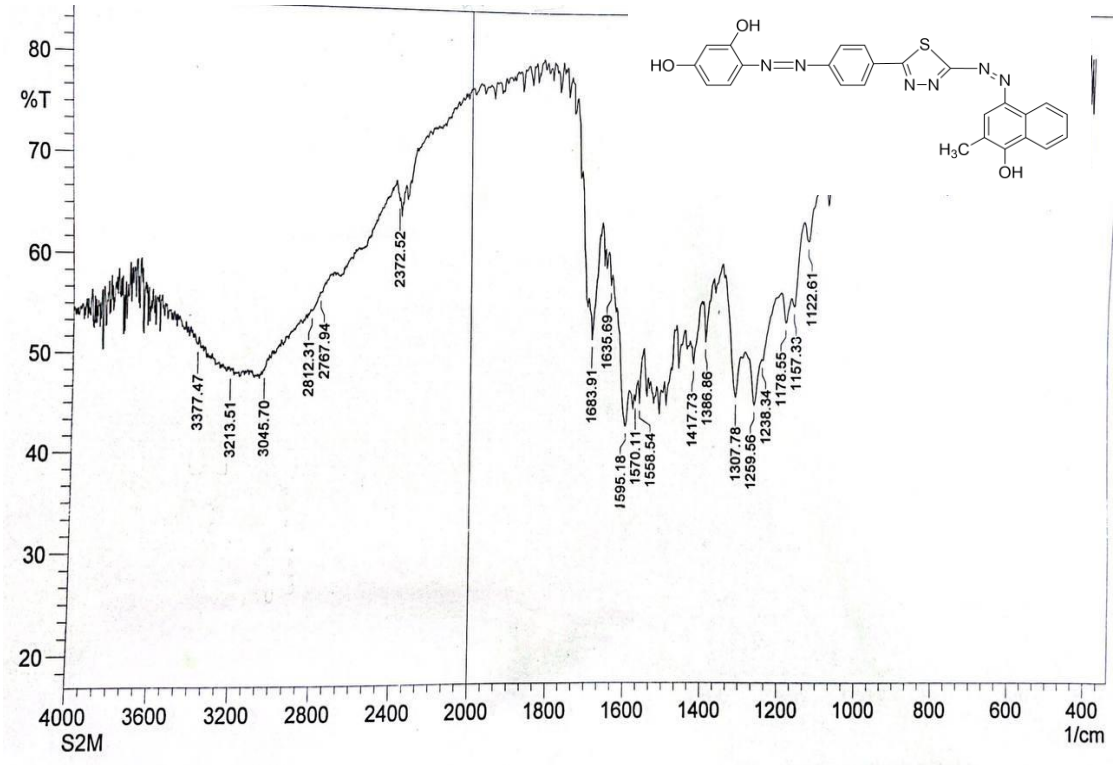


Figure 3-53: F.T-IR spectrum of compound [S19]

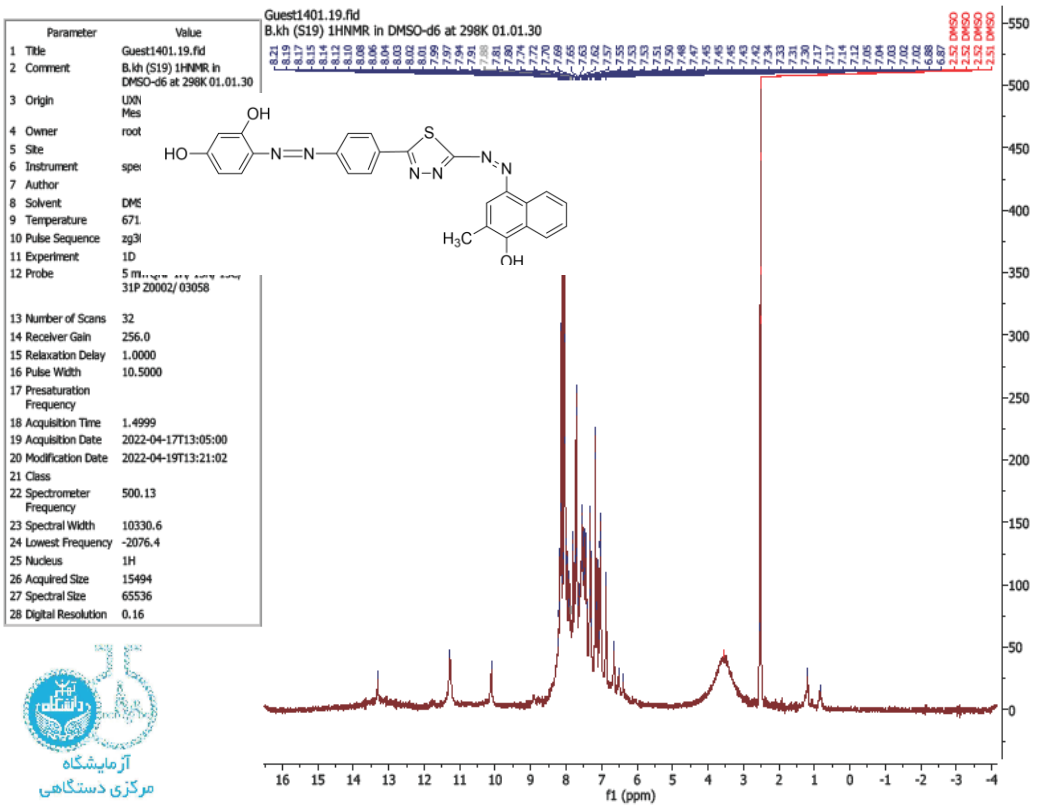
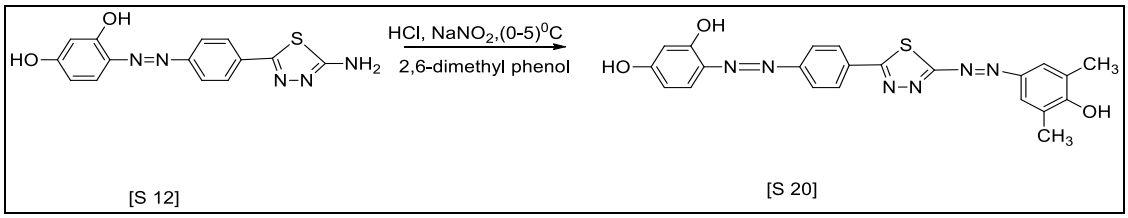


Figure3-54: ^{1}H-NMR spectrum of compound [S19]

Compound [S20]:



Equation 3-20: Synthesis of Compound [S20]

The FTIR spectrum of compound [S20] exhibited absorption band at (1429) cm^{-1} for N=N and disappearance absorption band at (3327,3221) cm^{-1} for NH_2

^{1}H-NMR(500MHz,DMSO-d_6) :(δ, ppm) spectrum show appearance signal at 6.26-8.16 for (H,Ar-H) and signal at 1.19-2.2 for (H,CH_3) and disappearance signal of (H,NH_2) .

IR (ν, cm^{-1}): O-H (3063), C-H_{aliph} (2941), C=N (1602) , C=C_{Ar} (1506) ,N=N (1429).

^{1}H-NMR (δ, ppm): ,(6H,CH_3) :1.19 -2.2 , (9H,Ar-H) : 6.26-8.16

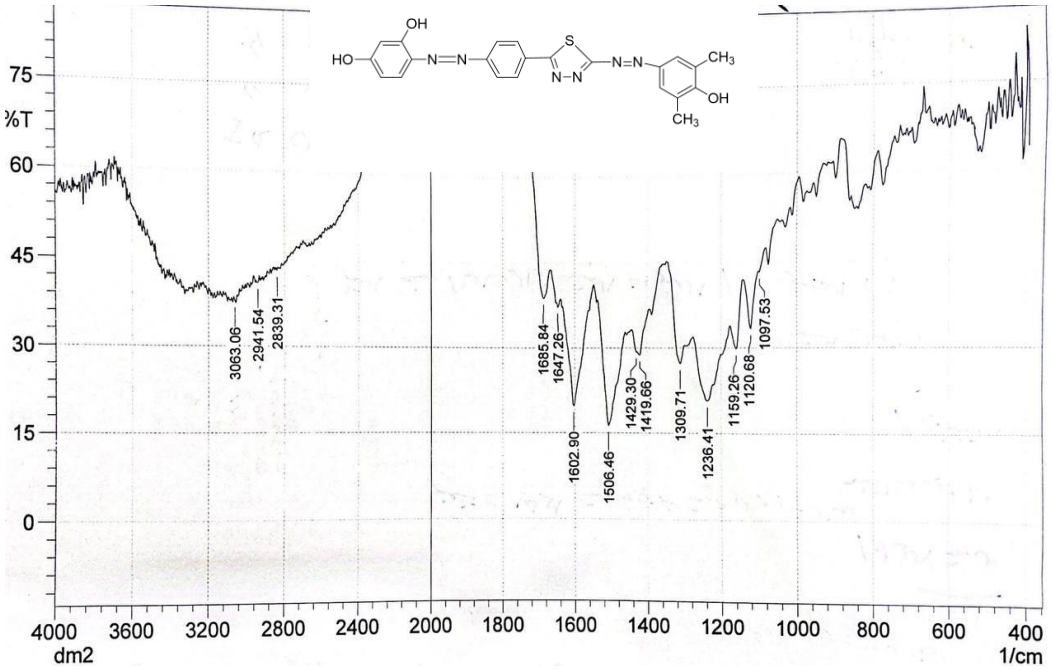


Figure3-55:F.T-IR spectrum of compound [S20]

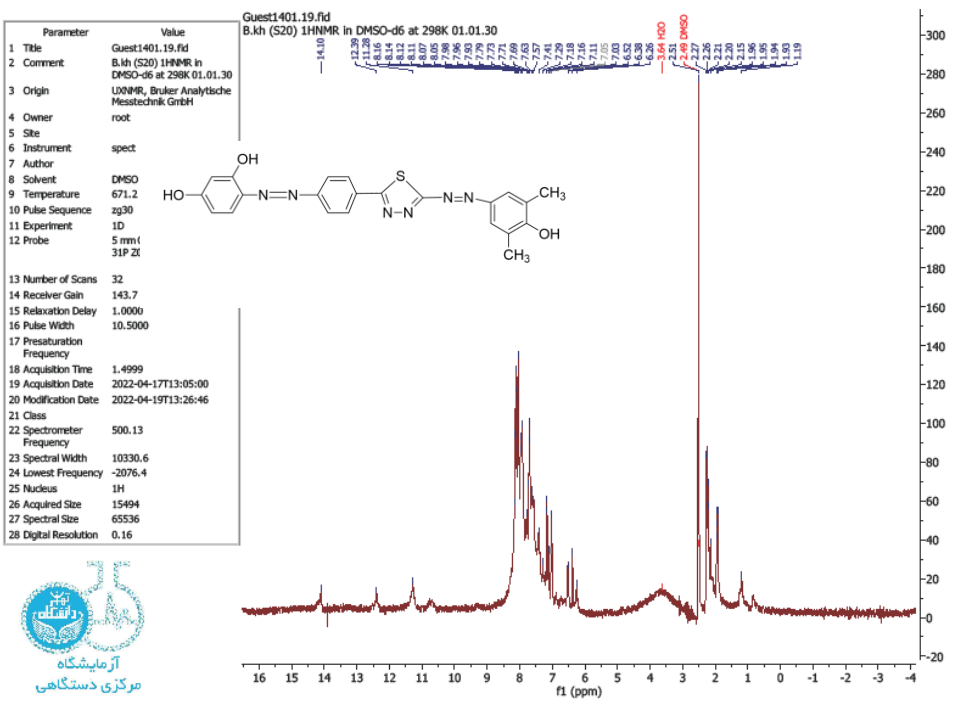
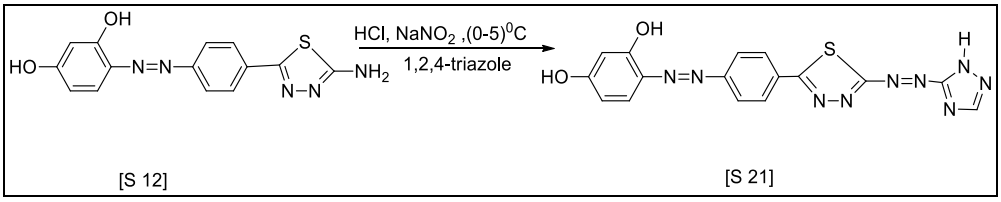


Figure3-56: ^{1}H-NMR spectrum of compound [S20]

Compound [S21]:



Equation 3-21: Synthesis of Compound [S21]

The FTIR spectrum of compound [S21] exhibited absorption band at (1425) cm^{-1} for N=N and disappearance absorption band at (3327,3221) cm^{-1} for NH_2

^{1}H-NMR(400MHz,DMSO-d_6) :(δ, ppm) spectrum show appearance signal at 6.51-8.1 for (H,Ar-H) and disappearance signal of (H,NH_2) .

IR (ν, cm^{-1}): O-H (3450),NH (3335),C-H $_{Ar}$,(3063),C=N (1599) , C=C_{Ar} (1510) , N=N (1425).

^{1}H-NMR (δ, ppm): (H,NH) : 6.9 (7 H,Ar-H) : 7.03-8.1

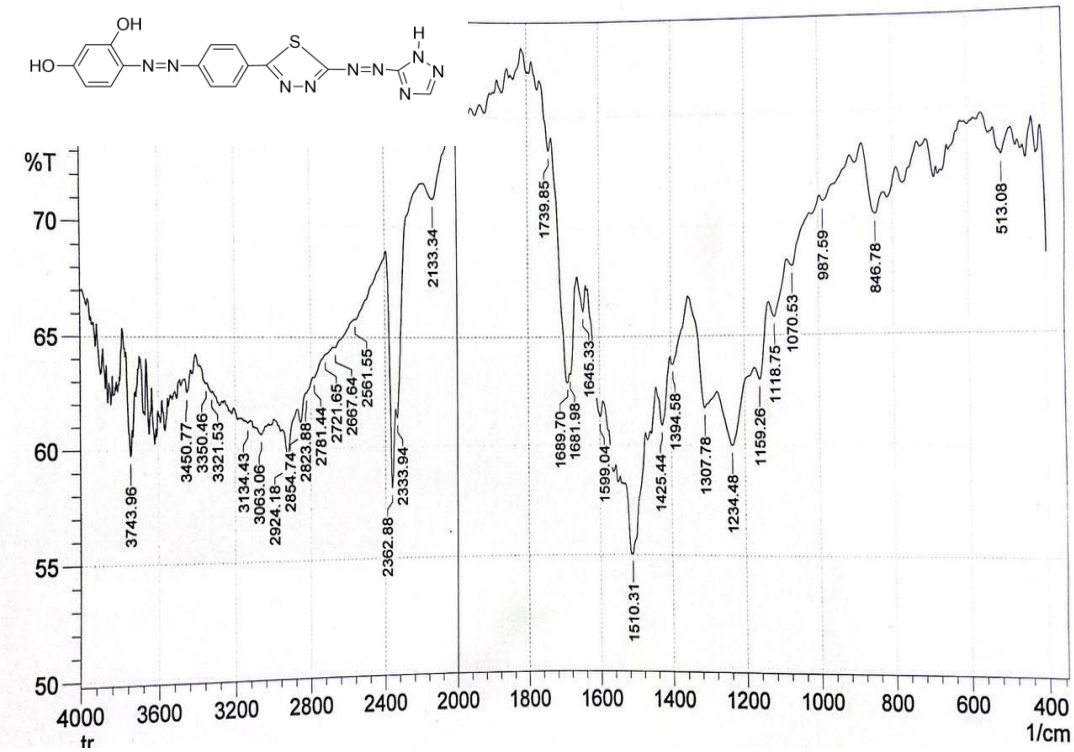


Figure3-57: FT-IR spectrum of compound [S21]

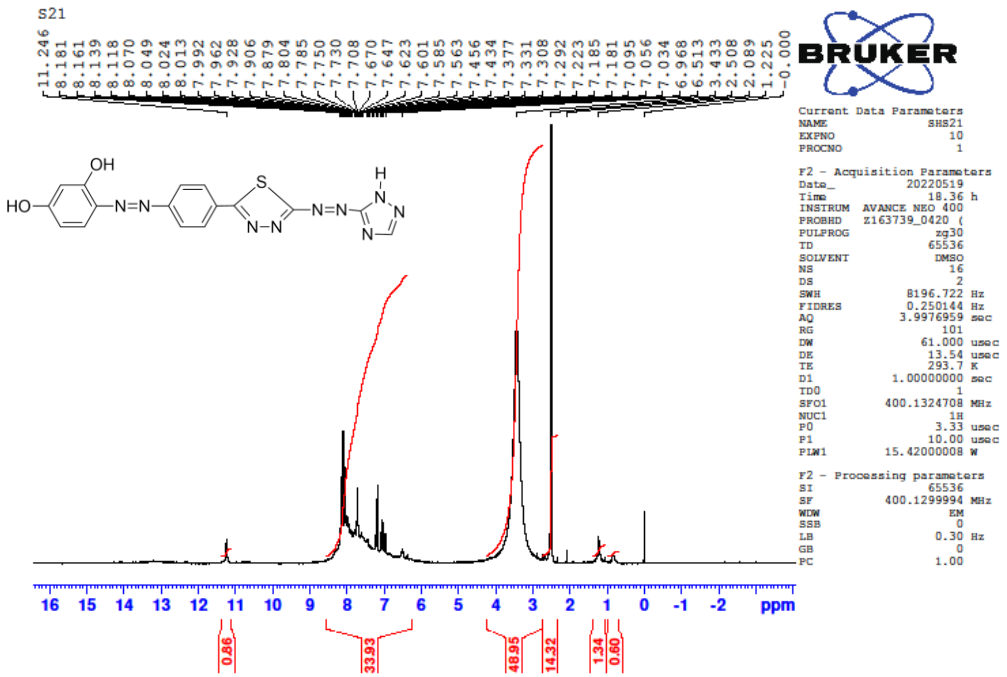
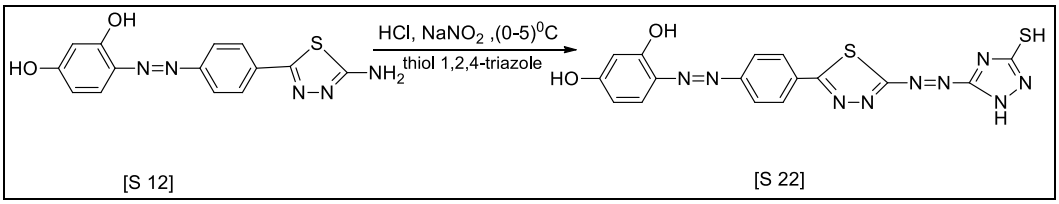


Figure3-58: ^{1}H-NMR spectrum of compound [S21]

Compound [S22]:



Equation 3-22: Synthesis of Compound [S22]

The FTIR spectrum of compound [S22] exhibited absorption band at (1419) cm^{-1} for N=N and disappearance absorption band at (3327,3221) cm^{-1} for NH_2

^{1}H-NMR(400MHz,DMSO-d_6) :(δ, ppm) spectrum show appearance signal at 6.4-8.3 for (H,Ar-H) and appearance signal of (H,NH) and disappearance signal of (H,NH_2) .

IR (ν, cm^{-1}): O-H (3360),C-H $_{Ar}$ (3105), C=N (1600) , C=C_{Ar} (1596), N=N (1419).

^{1}H-NMR (δ, ppm):(H,NH): 6.4, (7H,Ar-H) : 6.5-8.3

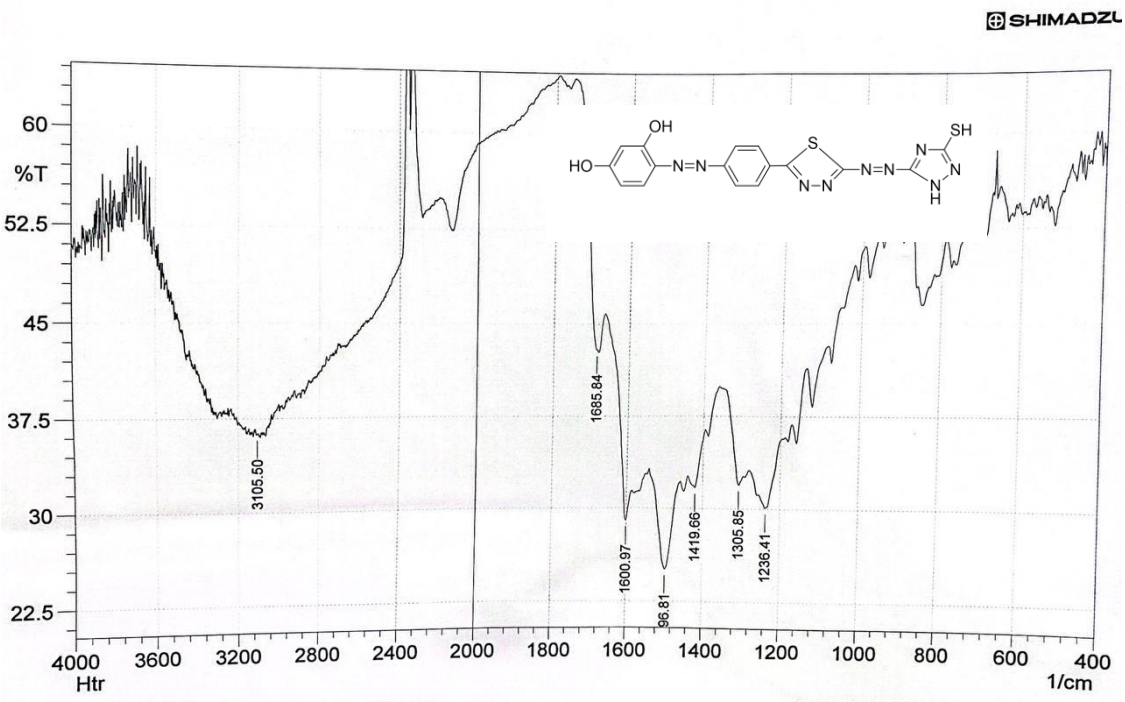


Figure3-59: FT-IR spectrum of compound [S22]

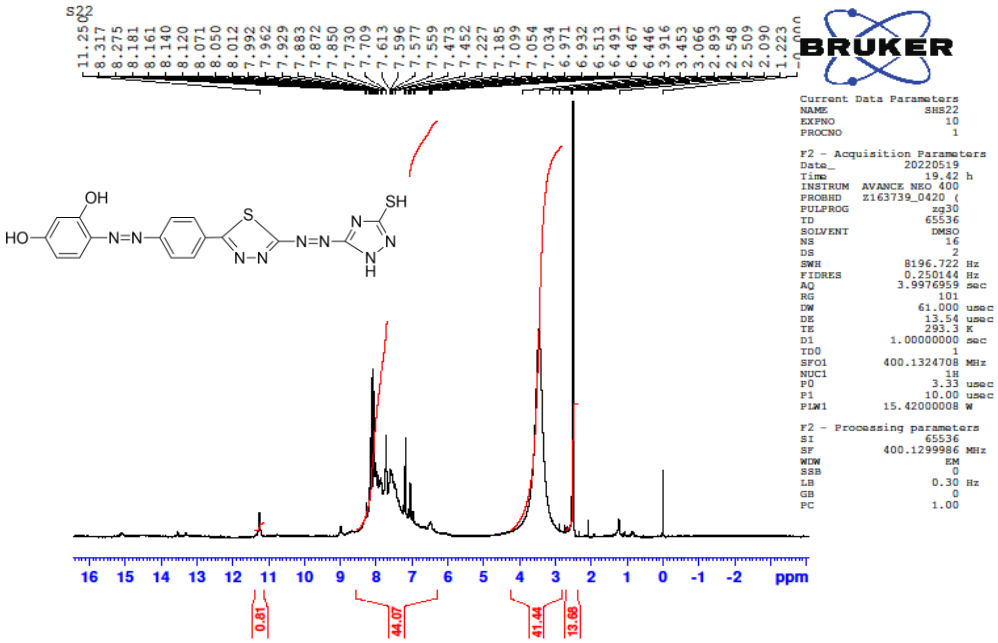
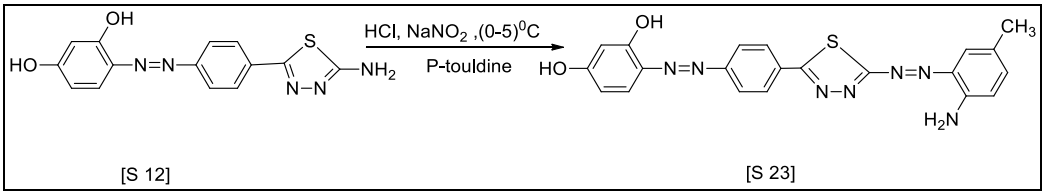


Figure3-60: ^{1}H-NMR spectrum of compound [S22]

Compound [S23]:



Equation 3-23: Synthesis of Compound [S23]

The FTIR spectrum of compound [S23] exhibited absorption band at (1452) cm^{-1} for N=N and disappearance absorption band at (3327,3221) cm^{-1} for NH_2

^{1}H-NMR(400MHz,DMSO-d_6) :(δ, ppm) spectrum show appearance signal at 6.8-8.14 for (H,Ar-H) and signal at 1.2 for (H,CH_3) and signal at 7.8 for (H,NH_2) .

IR (ν, cm^{-1}): O-H (3427),C-H $_{Ar}$ (3134),C-H $_{aliph}$ (2993),C=N (1602) , C=C_{Ar} (1491), N=N (1452).

^{1}H-NMR(δ,ppm):(3H,CH_3):1.2,(10H,Ar-H):6.8-8.14,(H,NH_2):7.8,(H,OH) :11.10 .

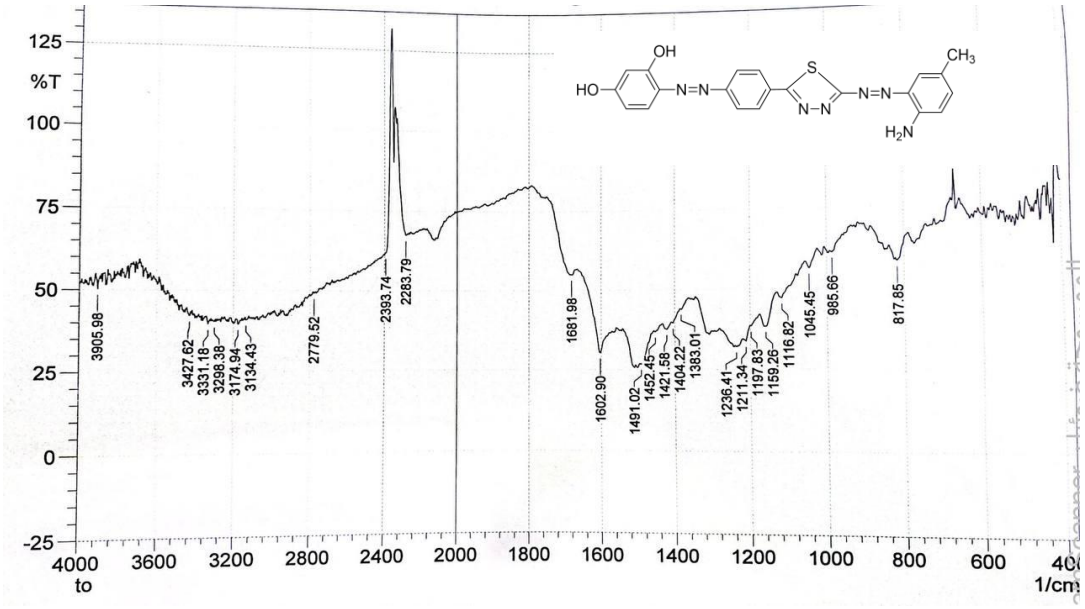


Figure3-61: FT-IR spectrum of compound [S23]

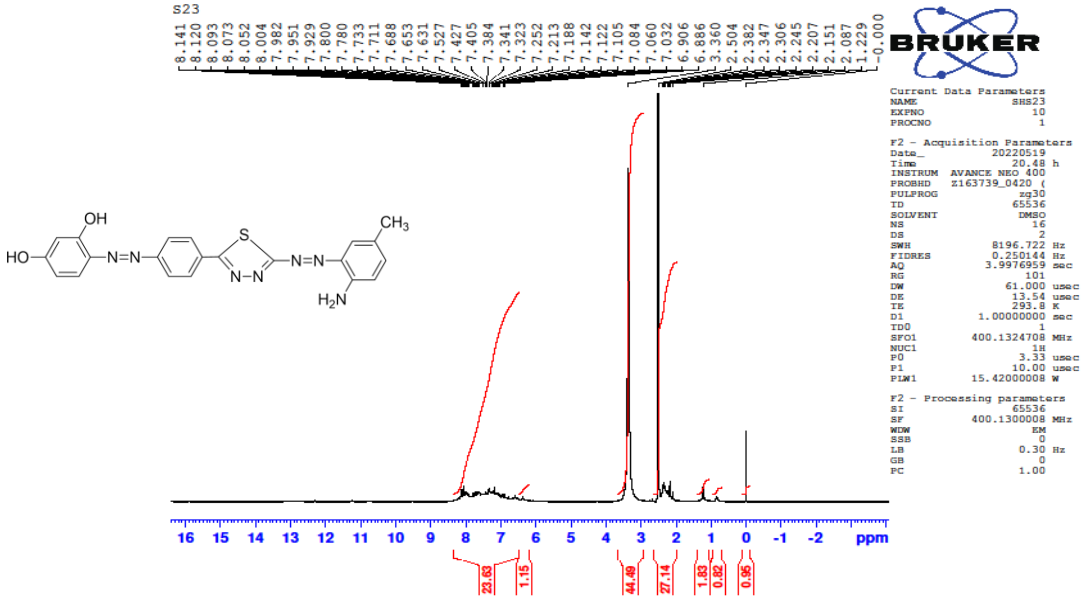
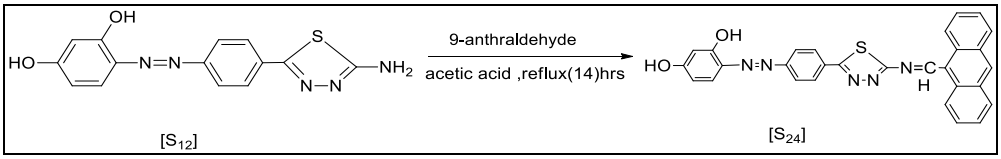


Figure3-62: ^{1}H-NMR spectrum of compound [S23]

3.8 Synthesis of compounds [S24-S29]:

In this reaction the amines react with aromatic aldehyde in nucleophilic reaction to form imine or azomethine bond ,this reaction called Schiff-base.

Compound [S24]:



Equation 3-24: Synthesis of the Compound[S24]

The FTIR spectrum showed disappearance absorption band of NH_2 at (3327,3221) and appearance absorption band of C- H_{aliph} at (2831) and absorption band of C=N $_{imine}$ at (1668) and absorption band of C=N at (1552).

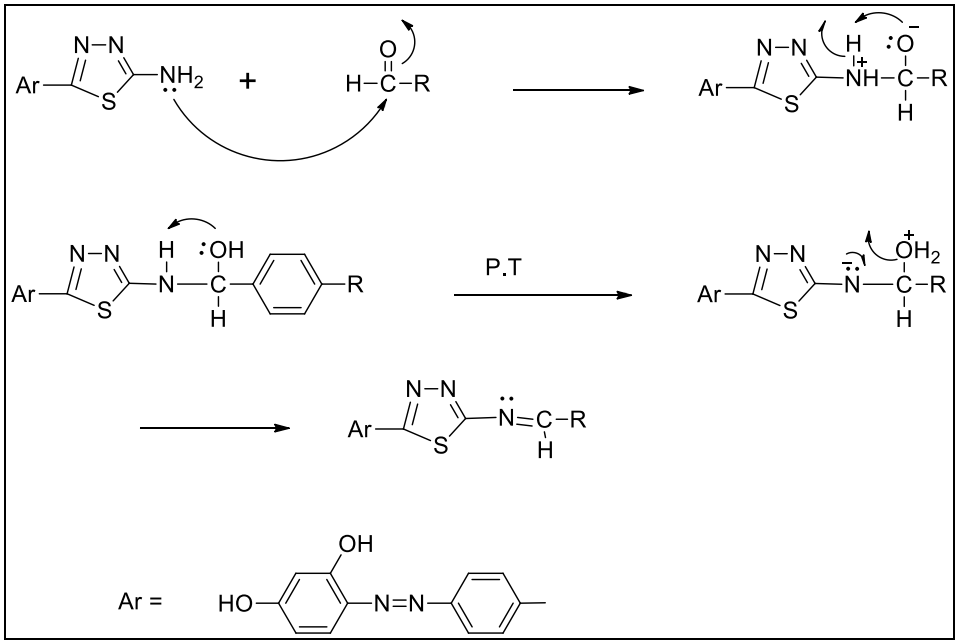
^{1}H-NMR(400MHz,DMSO-d_6) :(δ, ppm) spectrum exhibits disappearance signal for (H, NH_2)and appearance signal at 8.9 for (H,N=CH)
^{13}C-NMR (126MHz,DMSO-d_6) :(δ, ppm) spectrum showed signals at 123-140 for Ar-C and signal at 166 for C=N

IR (ν,cm^{-1}): C-H_{Ar} (3097) ,C-H_{alph} (2831) ,C=N $_{imine}$ (1668) ,C=C_{Ar} (1600), C=N (1552) ,N=N (1519) .

^{1}H-NMR (δ, ppm): (H,OH) 4.37,(H,Ar-H) 6.3-8.2 ,(H,N=CH) :8.9

^{13}C-NMR (δ, ppm) :123-140 for Ar-C,166 for C=N

The mechanism of synthesis schiff base is shown below [(94)]:



Scheme 3-6 : Mechanism of synthesis compounds [S24-S29]

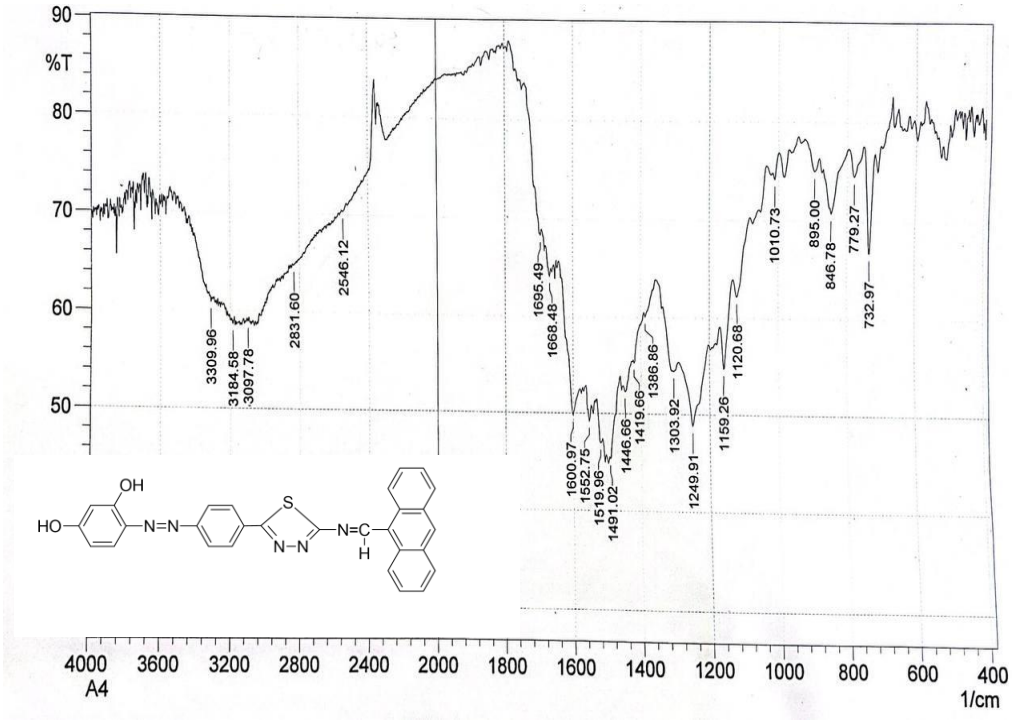


Figure3-63: FT-IR spectrum of compound [S24]

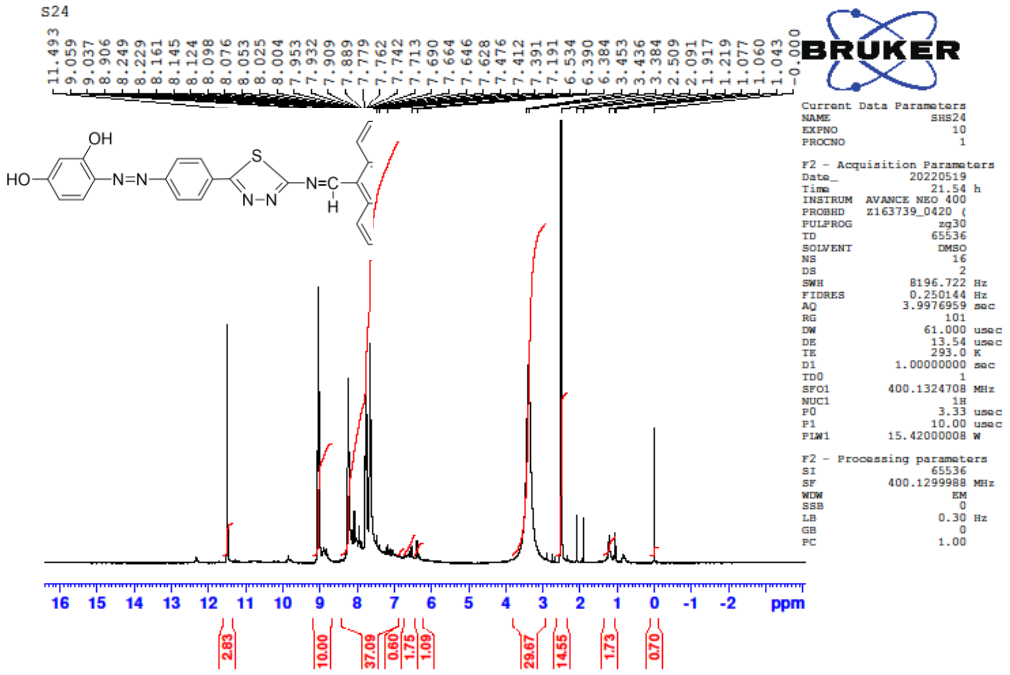


Figure3-64: ^{1}H-NMR spectrum of compound [S24]

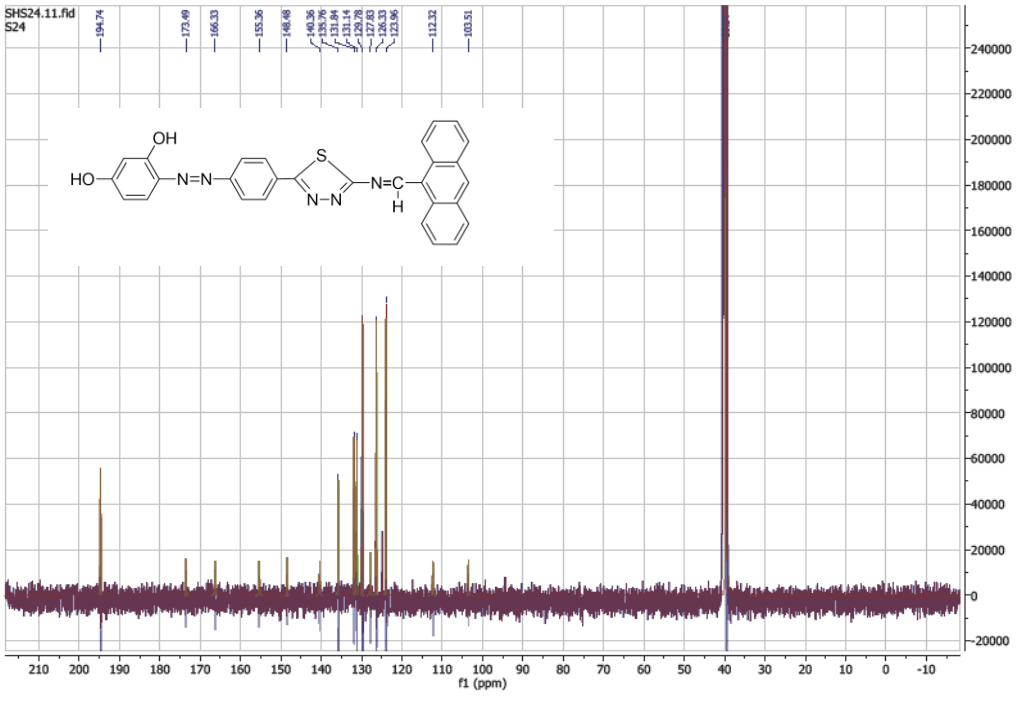
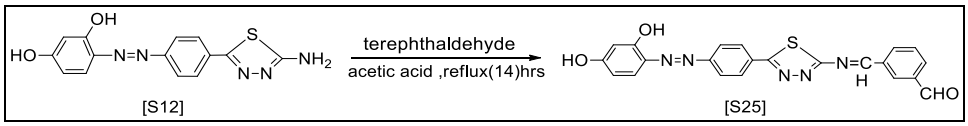


Figure3-65: ^{13}CNMR spectrum of compound [S24]

Compound [S25]:



[S12] [S25]

Equation 3-25: Synthesis of the Compound[S25]

The FTIR spectrum showed disappearance absorption band of NH_2 at (3327,3221) and appearance absorption band of C=N $_{imine}$ at (1602) and absorption band of C=N at (1533) .

^{1}H-NMR(400MHz,DMSO-d_6) :(δ, ppm) spectrum exhibits disappearance signal for (H, NH_2)and appearance signal at 8.12 for (H, N=CH) and signal for(H,CHO)

IR (ν,cm^{-1}): C-H_{Ar} (3155) ,C=$O_{aldehyde}$ (1697) , C=N $_{imine}$ (1602) , C=C_{Ar} (1570) ,C=N (1533) , N=N (1521) .

^{1}H-NMR (δ,ppm): (H,OH)5.86 ,(H,Ar-H)7.1-8.0 ,(H,N=CH) 8.12,(H,CHO) :10

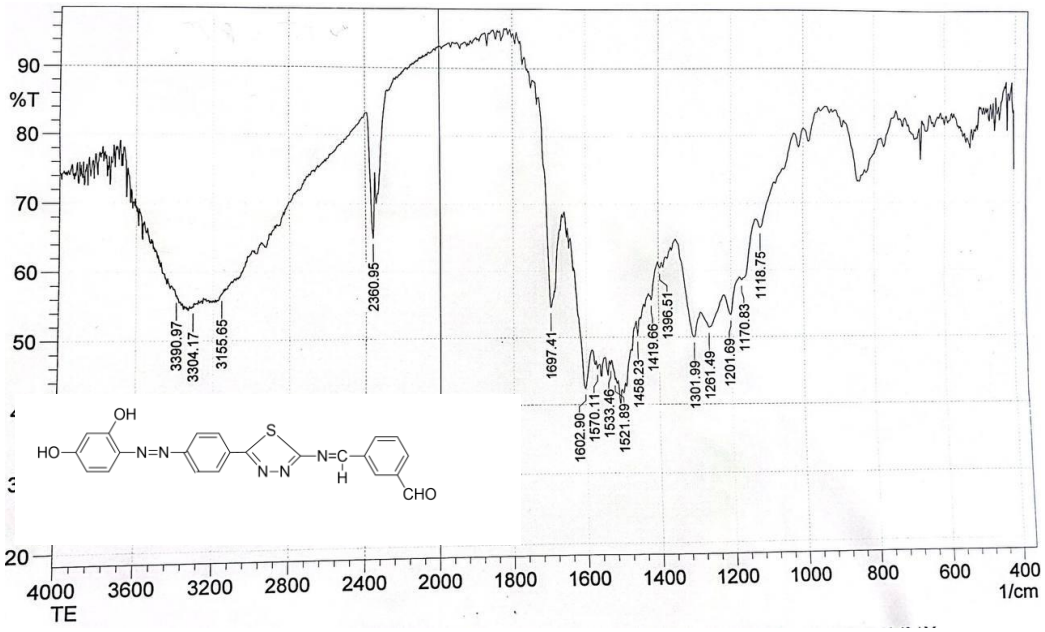


Figure3-66: FT-IR spectrum of compound [S25]

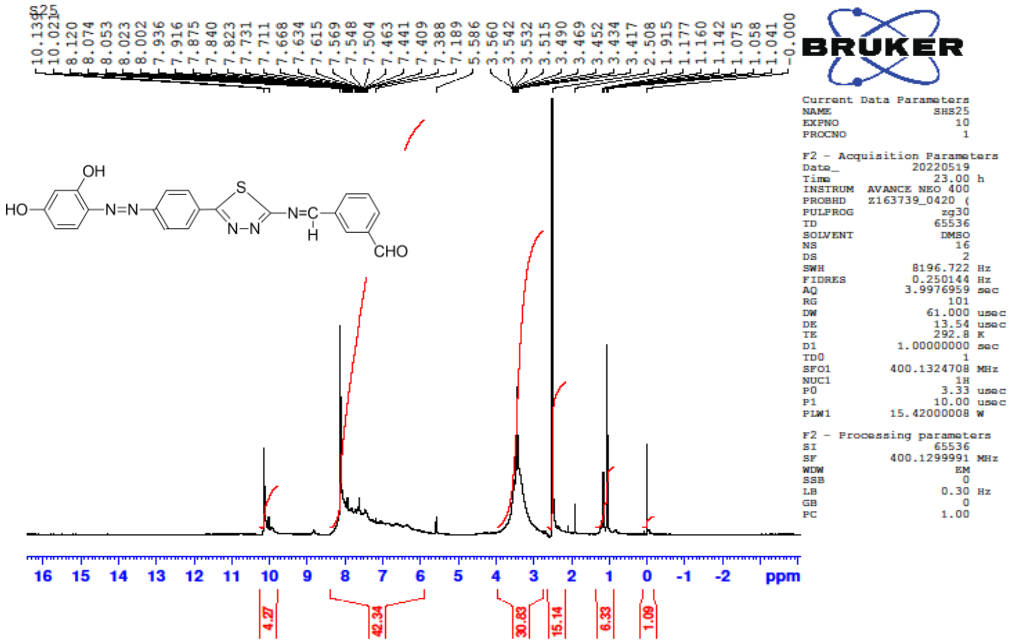
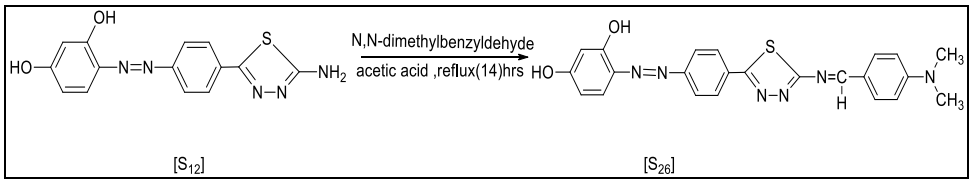


Figure3-67: ^{1}H-NMR spectrum of compound [S25]

Compound [S26]:



Equation 3-26: Synthesis of the Compound[S26]

The FTIR spectrum show disappearance absorption band of NH_2 at (3327,3221) and appearance absorption band of C- H_{aliph} at (2885) and absorption band of $C=N_{imine}$ at (1653) and absorption band of C=N at (1508).

^{1}H-NMR(400MHz,DMSO-d_6) :(δ, ppm) spectrum exhibits disappearance signal for (H, NH_2)and appearance signal at 8.12 for (H, N=CH) and signal at 2.0-2.8 for (6H,2CH_3)

IR (ν,cm^{-1}): C-H_{Ar} (3134) ,C-H_{alph} (2885) , $C=N_{imine}$ (1653) ,$C=C_{Ar}$ (1533) ,C=N (1508) , N=N (1491) .

^{1}H-NMR (δ, ppm):(6H,2CH_3) 2.0-2.8 , (H,Ar-H) 6.3-8.0 ,(H,N=CH) 8.12

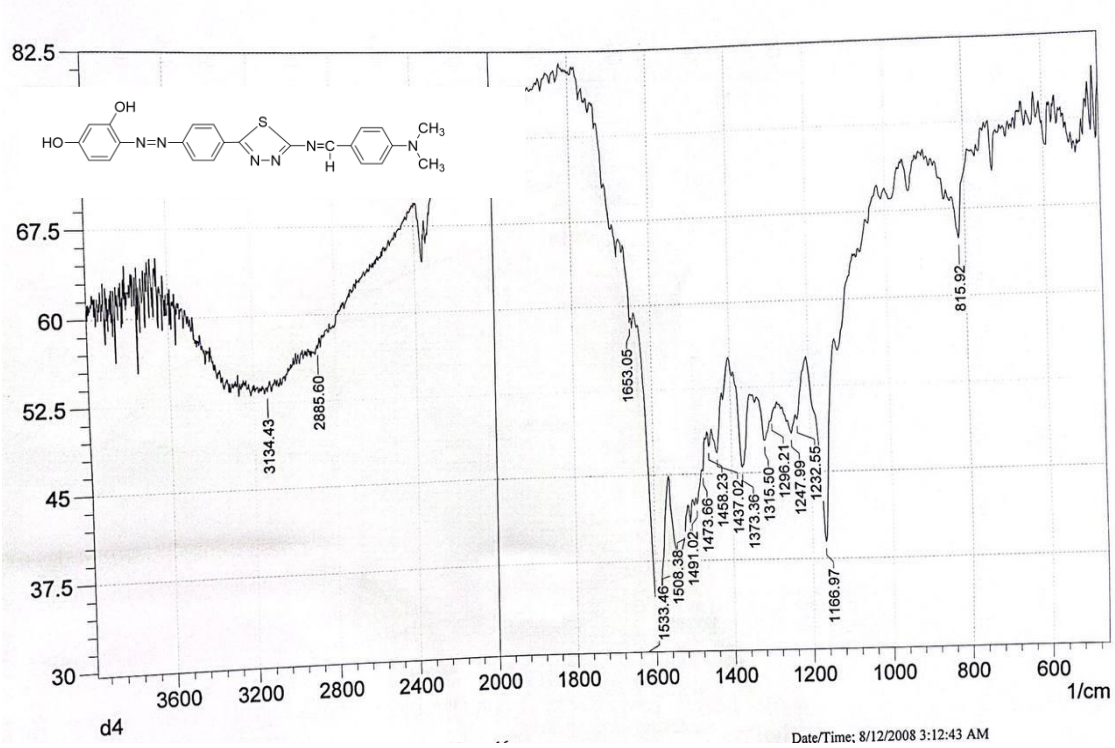


Figure3-68: FT-IR spectrum of compound [S26]

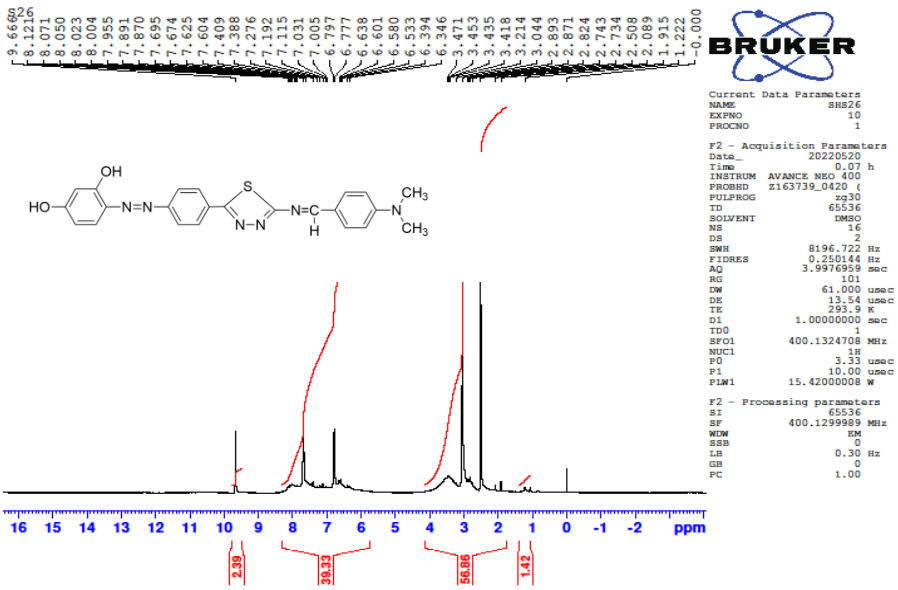
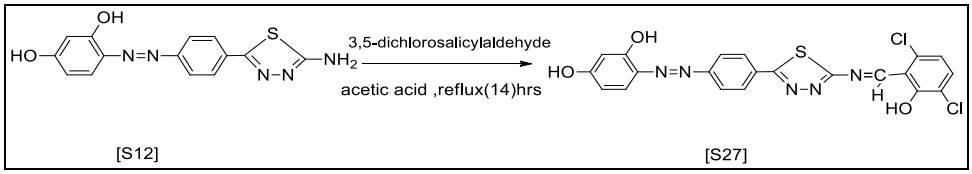


Figure3-69:

^{1}H-NMR spectrum of compound [S26]

Compound [S27]:



Equation 3-27: Synthesis of the Compound[S27]

The FTIR spectrum show disappearance absorption band of NH_2 at (3327,3221) and appearance absorption band of C- H_{aliph} at (2928) and absorption band of C=N $_{imine}$ at (1681) and absorption band of C=N at (1560).
^{1}H-NMR(400MHz,DMSO-d_6) :(δ, ppm) spectrum exhibits disappearance signal for (H, NH_2)and appearance signal at 8.1 for (H, N=CH)

^{13}C-NMR (126MHz,DMSO-d_6) :(δ,ppm) spectrum show signals at 118-156 for Ar-C and signal at 167 for C=N

IR (ν,cm^{-1}): C-H_{Ar} (3074) , C-H $_{alph}$ (2928) ,C=N $_{imine}$ (1681) , C=C_{Ar} (1600), C=N (1560) , N=N (1500) ,C-Cl (850) .

^{1}H-NMR (δ, ppm): (H,Ar-H) 6.4-8.0,(H,N=CH)8.1

^{13}C-NMR (δ, ppm) : 118-156 for Ar-C,167 for C=N .

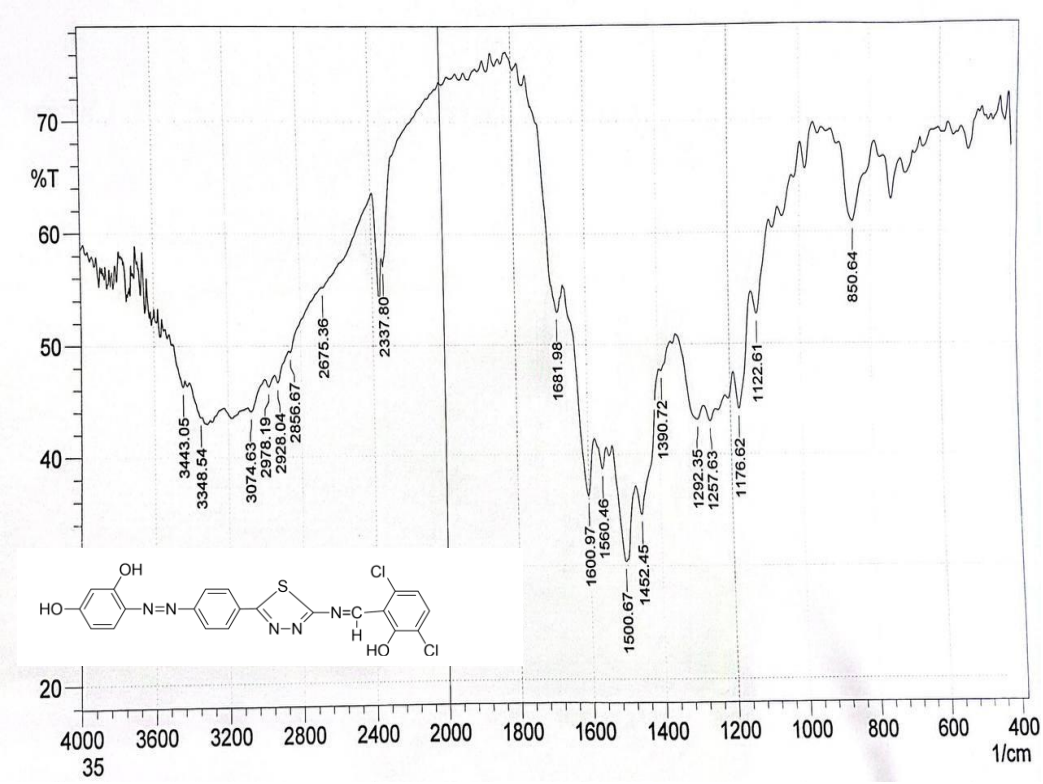


Figure3-70: FT-IR spectrum of compound [S27]

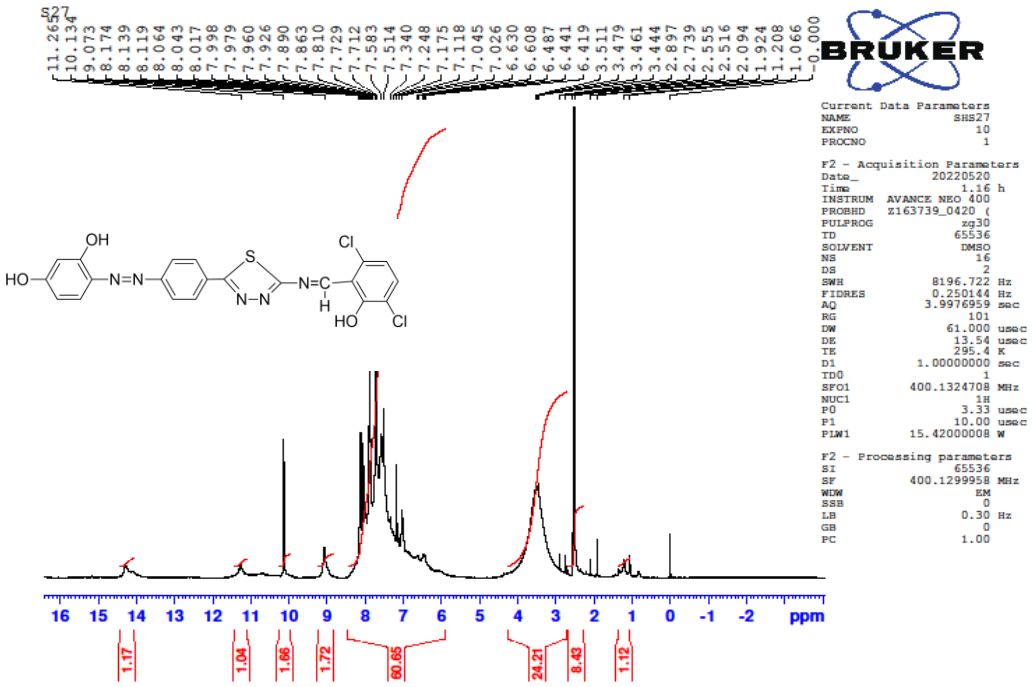


Figure3-71: ^{1}H-NMR spectrum of compound [S27]

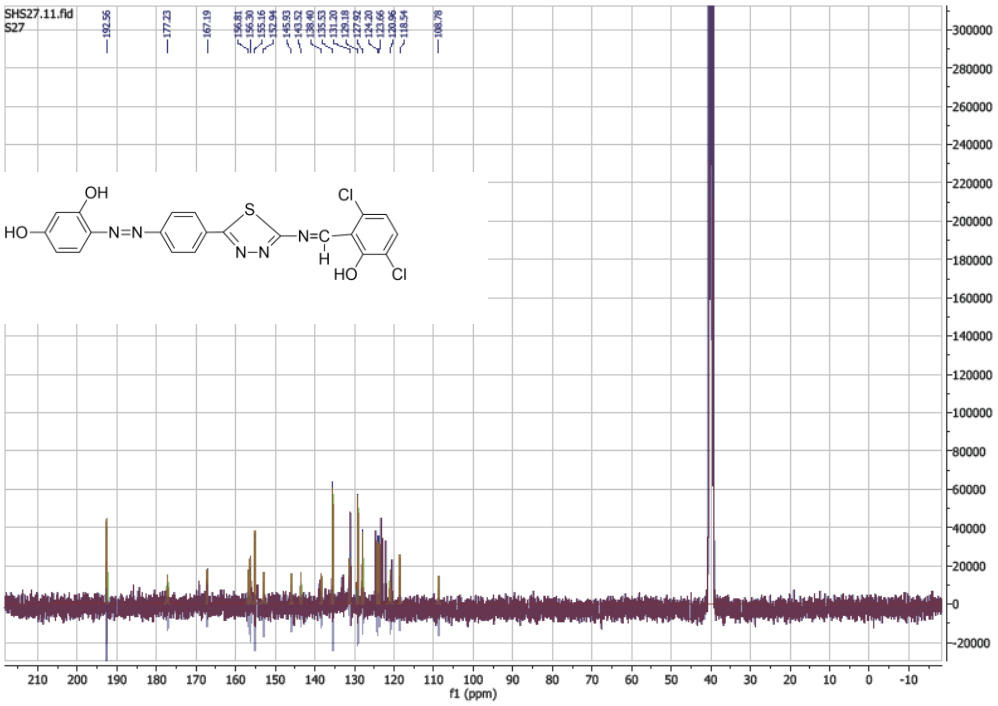
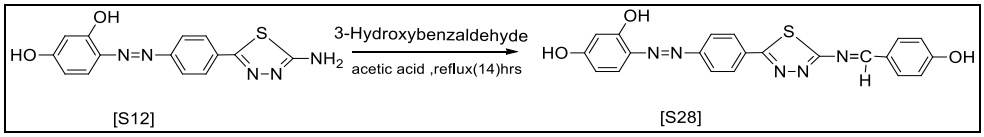


Figure3-72: ^{13}CNMR spectrum of compound [S27]

Compound [S28]:



Equation 3-28: Synthesis of the Compound[S28]

The FTIR spectrum show disappearance absorption band of NH_2 at (3327,3221) and appearance absorption band of C- H_{aliph} at (2843) and absorption band of C=N_{imine} at (1681) and absorption band of C=N at (1560).

^{1}H-NMR(400MHz,DMSO-d_6) :(δ, ppm) spectrum exhibits disappearance signal for (H, NH_2)and appearance signal at 8.5 for (H, N=CH)

^{13}C-NMR (126MHz,DMSO-d_6) :(δ, ppm) spectrum show signals at 114-158 for Ar-C and signal at 167 for C=N

IR (ν,cm^{-1}): C-H_{Ar} (3070) , C-H $_{alph}$ (2843) ,C=N_{imine} (1681) ,C=C_{Ar} (1599) ,C=N (1560) , N=N (1539) .

^{1}H-NMR (δ, ppm): (H,Ar-H) 6.4 -8.1 ,(H,N=CH) 8.5

^{13}C-NMR (δ, ppm) :114-158 for Ar-C,167 for C=N

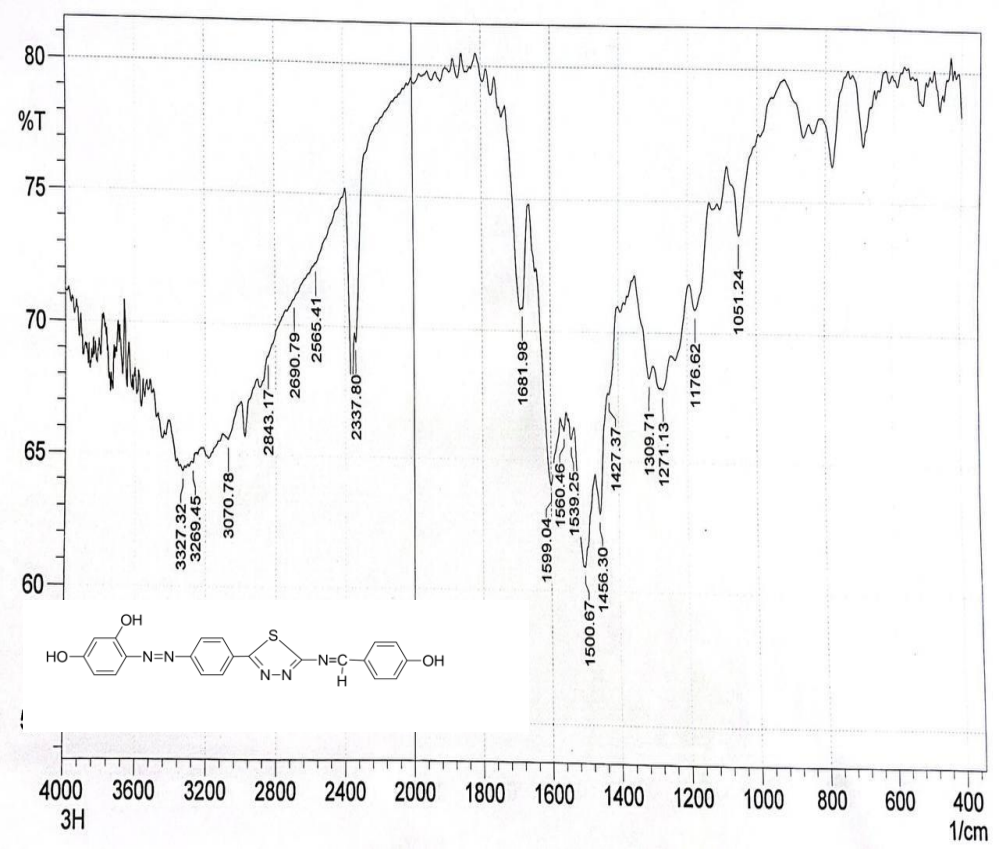


Figure3-73: FT-IR spectrum of compound [S28]

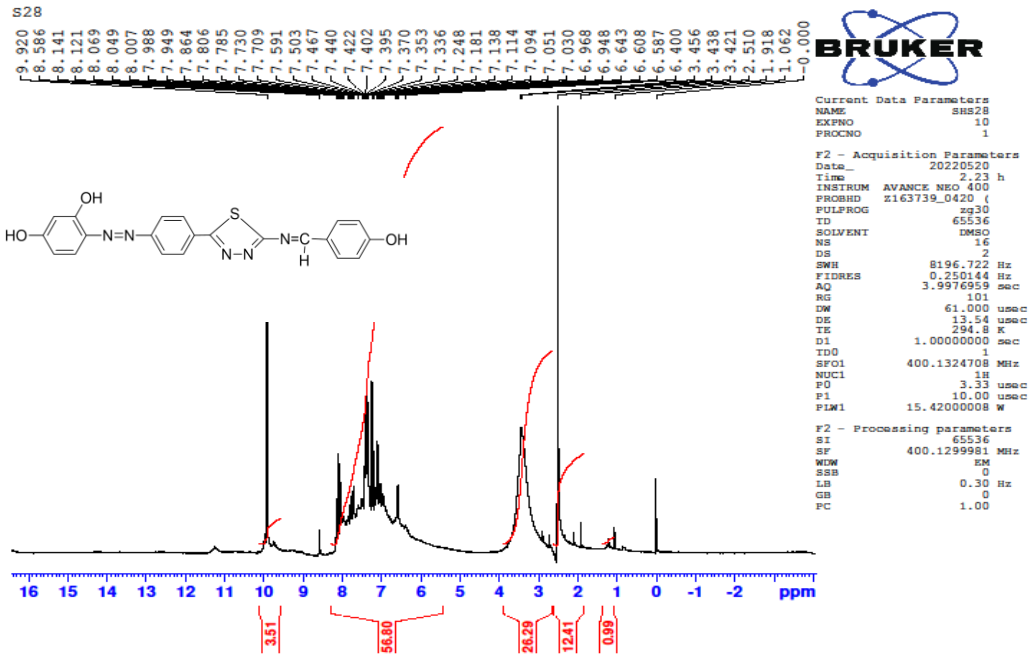


Figure 3-74: ^{1}H-NMR spectrum of compound [S28]

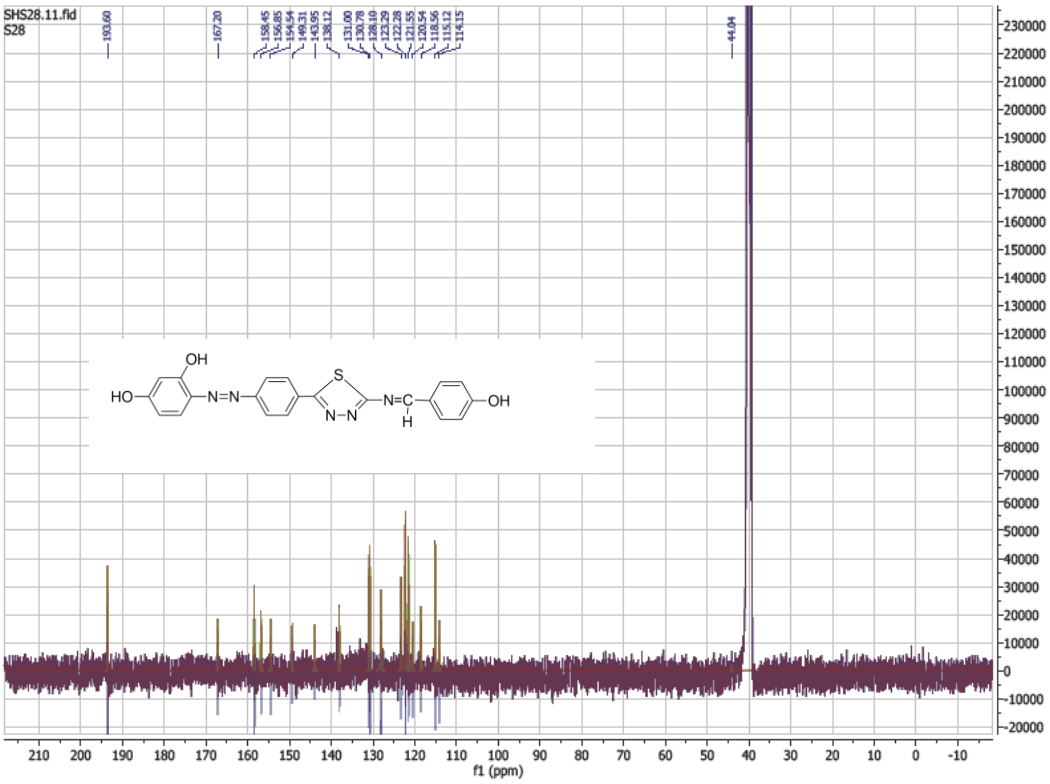
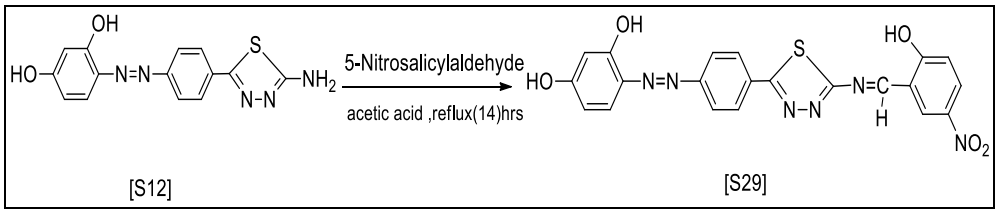


Figure3-75: ^{13}CNMR spectrum of compound [S28]

Compound [S29]:



Equation 3-29: Synthesis of the Compound[S29]

The FTIR spectrum show disappearance absorption band of NH_2 at (3327,3221) and appearance absorption band of C- H_{aliph} at (2922) and absorption band of $C=N_{imine}$ at (1681) and absorption band of C=N at (1572).

^{1}H-NMR(400MHz,DMSO-d_6) :(δ, ppm) spectrum exhibits disappearance signal for (H, NH_2)and appearance signal at 8.4 for (H, N=CH)
IR (ν,cm^{-1}): C-H_{Ar} (3080) ,C-H $_{alph}$ (2922) , C=N $_{imine}$ (1681) , C=C_{Ar} (1600), C=N (1572) , N=N (1492) , NO_2 (1340).

^{1}H-NMR (δ, ppm): (H,Ar-H) 6.91-8.3 ,(H,N=CH) 8.4

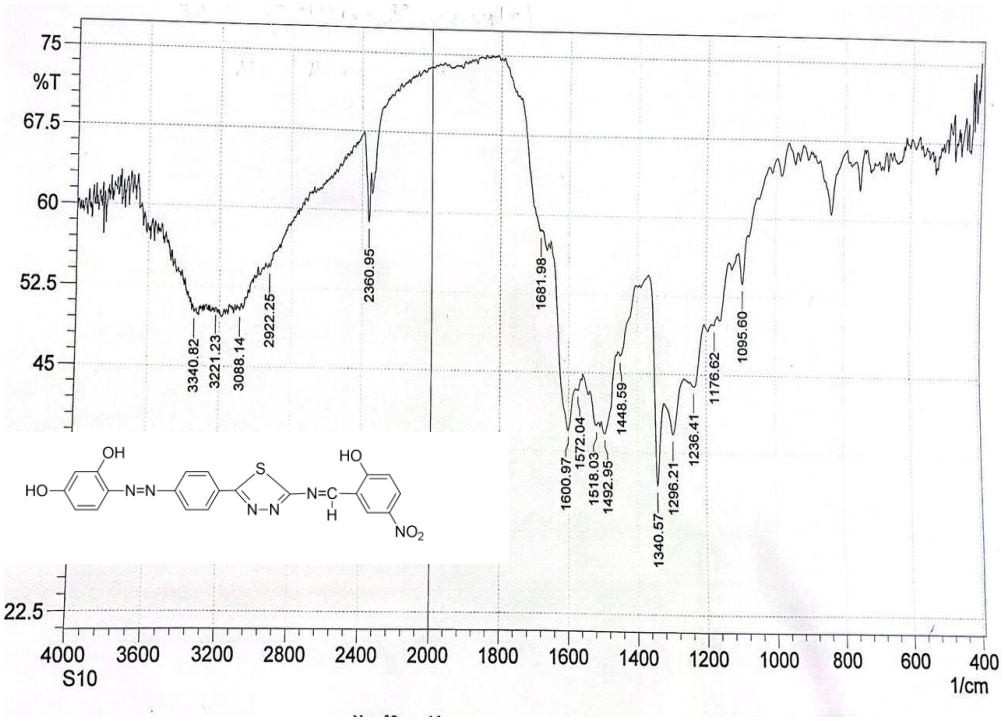


Figure 3-76: FT-IR spectrum of compound [S29]

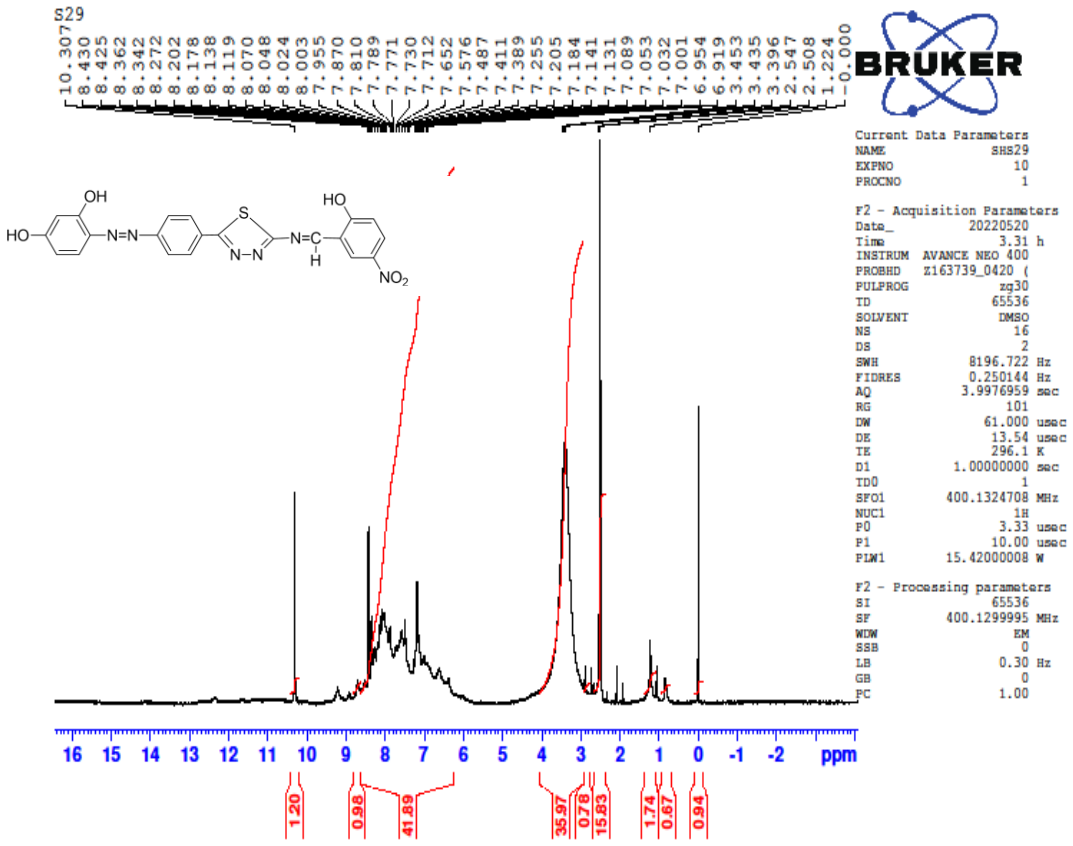


Figure 3-77: ^{1}H-NMR spectrum of compound [S29]

Table 1: C.H.N.S data of the prepared compounds [S1-S29]

Comp. NO	C% Calculation	C% Measure	H% Calculation	H% Measure	N Calculation %	N% Measure	S% Calculation	S% Measure
[S1]	54.545	53.22	3.496	3.94	21.70	9.23	-	-
[S2]	62.937	62.34	4.895	4.01	19.58	9.13	-	-
[S3]	57.352	57.81	4.411	4.82	20.588	20.11	-	-
[S4]	57.954	57.13	3.409	3.03	19.88	19.46	-	-
[S5]	62.682	61.48	3.482	3.94	17.412	17.01	-	-
[S6]	56.124	56.87	3.340	2.98	21.82	21.46	-	-
[S7]	57.627	56.93	3.954	4.22	19.77	19.33	-	-
[S8]	60.355	60.77	4.142	4.67	16.56	16.93	-	-
[S9]	64.285	63.88	4.761	4.70	16.666	16.54	-	-
[S10]	61.445	61.92	4.347	4.84	17.391	16.99	-	-
[S11]	65.142	64.69	5.142	5.63	16	16.33	-	-
[S12]	53.674	53.00	3.514	3.18	22.364	21.95	10.223	9.69
[S13]	56.25	55.87	3.571	3.45	18.75	18.70	7.142	7.12
[S14]	55.299	55.74	3.225	3.11	19.354	18.55	7.373	7.17
[S15]	55.299	55.69	3.255	3.05	19.354	19.28	7.373	7.34
[S16]	55.299	55.78	3.255	3.48	19.354	19.17	7.373	7.40
[S17]	61.538	61.12	3.418	3.33	17.948	17.66	6.837	6.80
[S18]	61.538	61.84	3.418	3.42	17.948	17.85	6.837	6.76
[S19]	62.240	61.89	3.734	3.26	17.427	17.31	6.639	6.60
[S20]	59.192	58.95	4.035	3.87	18.834	18.67	7.174	7.07
[S21]	48.85	48.54	2.79	2.69	32.06	32.00	8.14	8.33
[S22]	45.17	45.44	2.58	2.48	29.64	29.58	15.05	14.98
[S23]	58.46	48.28	3.94	3.85	22.73	22.66	7.42	7.58
[S24]	69.46	69.18	3.79	3.65	13.97	14.26	6.38	6.57
[S25]	61.53	61.39	3.49	3.78	16.31	15.94	7.45	7.11
[S26]	62.16	61.85	4.50	4.11	18.91	18.56	7.20	6.89
[S27]	51.85	51.45	3.67	3.48	14.40	14.94	6.58	6.11
[S28]	60.43	60.09	3.59	3.88	16.78	16.32	7.67	7.85
[S29]	54.54	54.81	3.03	2.79	18.18	18.55	6.92	7.43

Table 2: Solubility of the prepared compounds [S1-S29]

Compound Name	Solvents					
	H_2O	DMSO	DCM	Diethyl ether	Ethanol	Hexane
[S1]	-	+	-	-	+	-
[S2]	-	+	+	-	+	δ
[S3]	-	+	+	+	+	-
[S4]	-	+	+	+	-	-
[S5]	-	+	+	-	-	-
[S6]	-	+	+	-	+	-
[S7]	-	+	+	+	δ	-
[S8]	-	+	+	-	-	-
[S9]	-	+	δ	+	-	δ
[S10]	-	+	+	-	-	-
[S11]	-	+	+	δ	-	+
[S12]	-	+	+	-	+	-
[S13]	-	+	δ	+	δ	δ
[S14]	-	+	+	-	-	-
[S15]	-	-	-	+	-	-
[S16]	-	+	-	-	δ	+
[S17]	-	+	+	+	δ	-
[S18]	-	+	δ	+	+	δ
[S19]	-	+	+	-	+	-
[S20]	-	+	+	+	-	+
[S21]	-	+	-	+	-	-
[S22]	-	+	+	δ	-	-
[S23]	-	+	+	+	+	-
[S24]	-	+	+	+	-	-
[S25]	-	+	+	-	δ	-
[S26]	-	+	-	+	δ	-
[S27]	-	+	+	-	+	δ
[S28]	-	+	+	-	+	+
[S29]	-	+	-	+	-	-

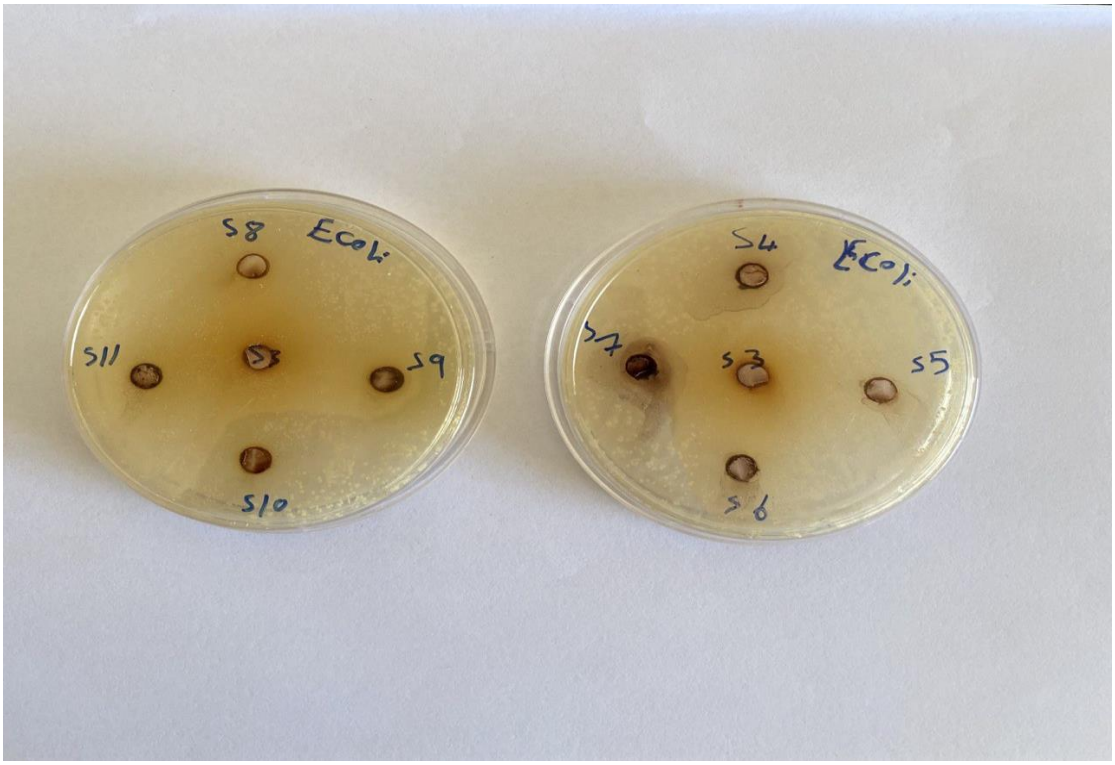
3.9 Biological Activity :

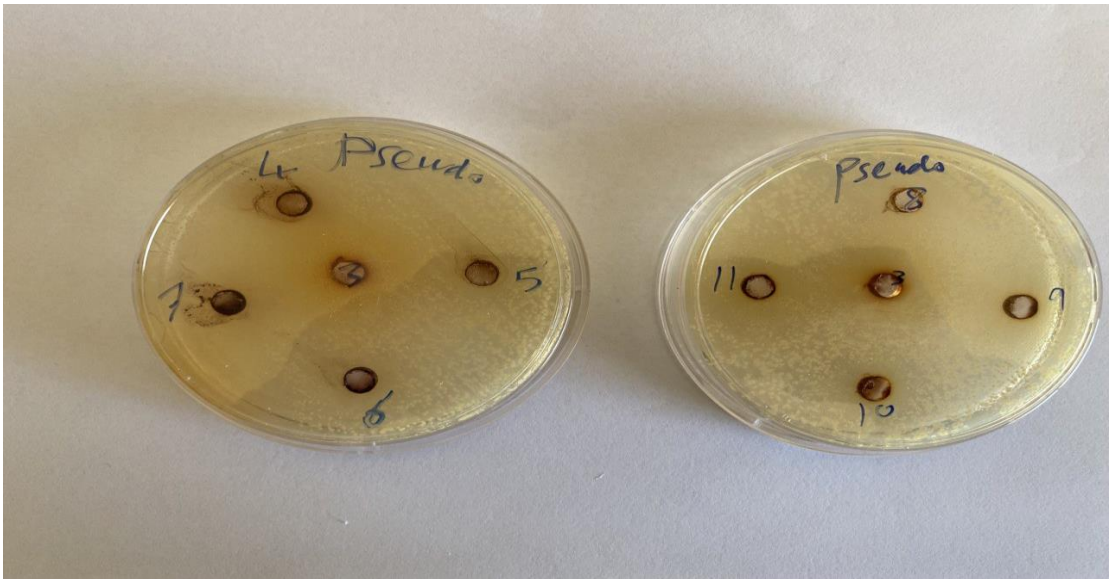
Biological activity of the synthesized compounds were evaluated by the agar disc-diffusion method against gram positive (*Staphylococcus epiderimidis* and *Staphylococcus aureus*) and gram negative (*Klips, pseudomonas*, and *E. coli*) bacteria. The concentration of the investigated chemicals was 10^{-3} M, with dimethyl sulphoxide (DMSO) serving as the solvent control. The findings of these studies indicate that [S3 and S8] have good anti-bacterial activity [30,27] because of their structural contact with specific bacteria's cell walls, which led to high inhibition,also [S4and S6] have good antibacterial on *Staphylococcus epiderimidis* .

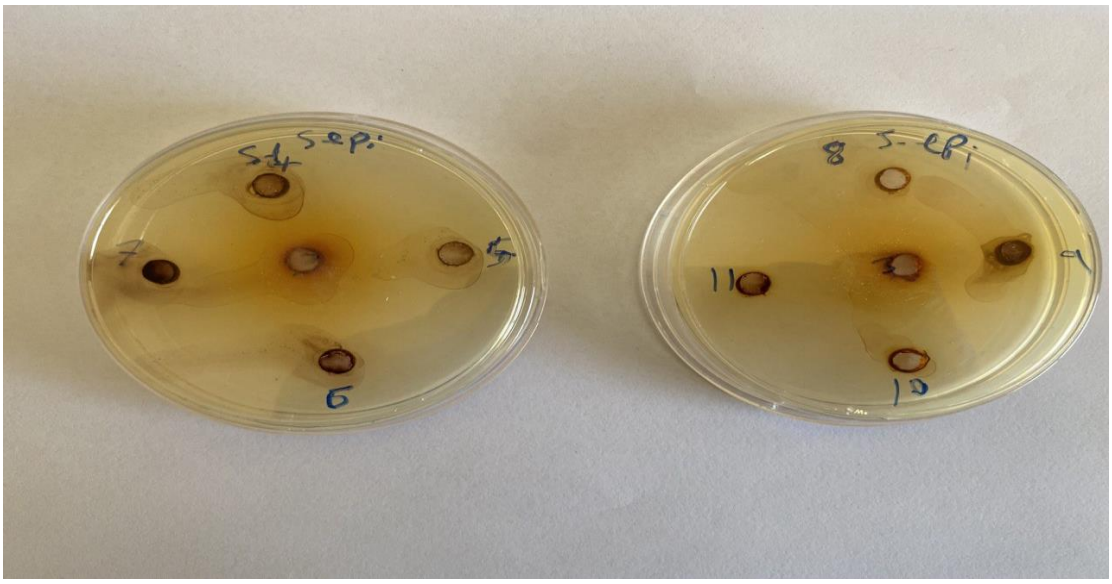
Table 3-3 summarizes the antibacterial activity of produced compounds [S3-S11] (figure78).

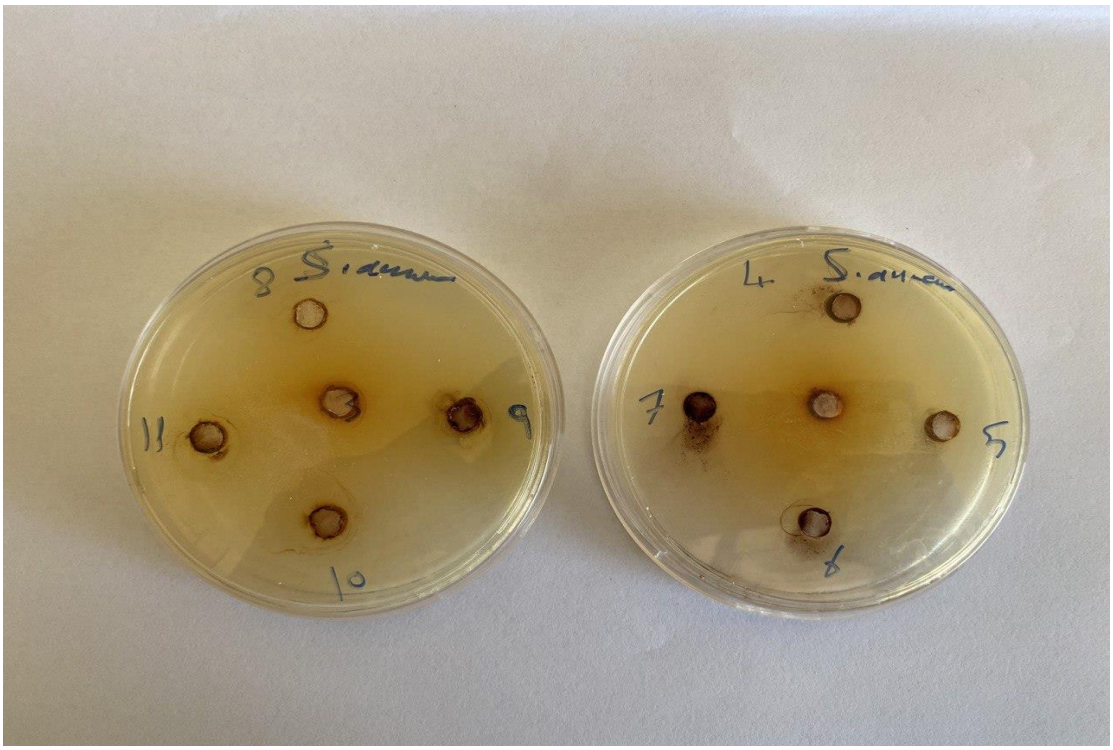
Table 3- 3: antibacterial activity of the compounds [S3-S11]

Compound number	*Staphylococcus epiderimidis*	*Staphylococcus aureus*	*E.coli*	*Klebsiella pneumoniae*	*pseudomonas*
[S3]	26	26	30	13	30
[S4]	25	14	20	10	20
[S5]	18	12	20	0	13
[S6]	25	13	13	9	15
[S7]	20	13	15	9	20
[S8]	0	11	26	12	27
[S9]	23	12	25	12	20
[S10]	13	15	13	11	18
[S11]	0	13	23	0	20


S8 Ecoli
S11
S9
S10
S4 Ecoli
S7
S3
S5
S6


Pseudo
Pseudo
8
11
9
10


5 S.ep.
7
5
6
8 S. ep.
11
9
10


3 S. aureus
11
9
10
4 S. aureus
7
5
8

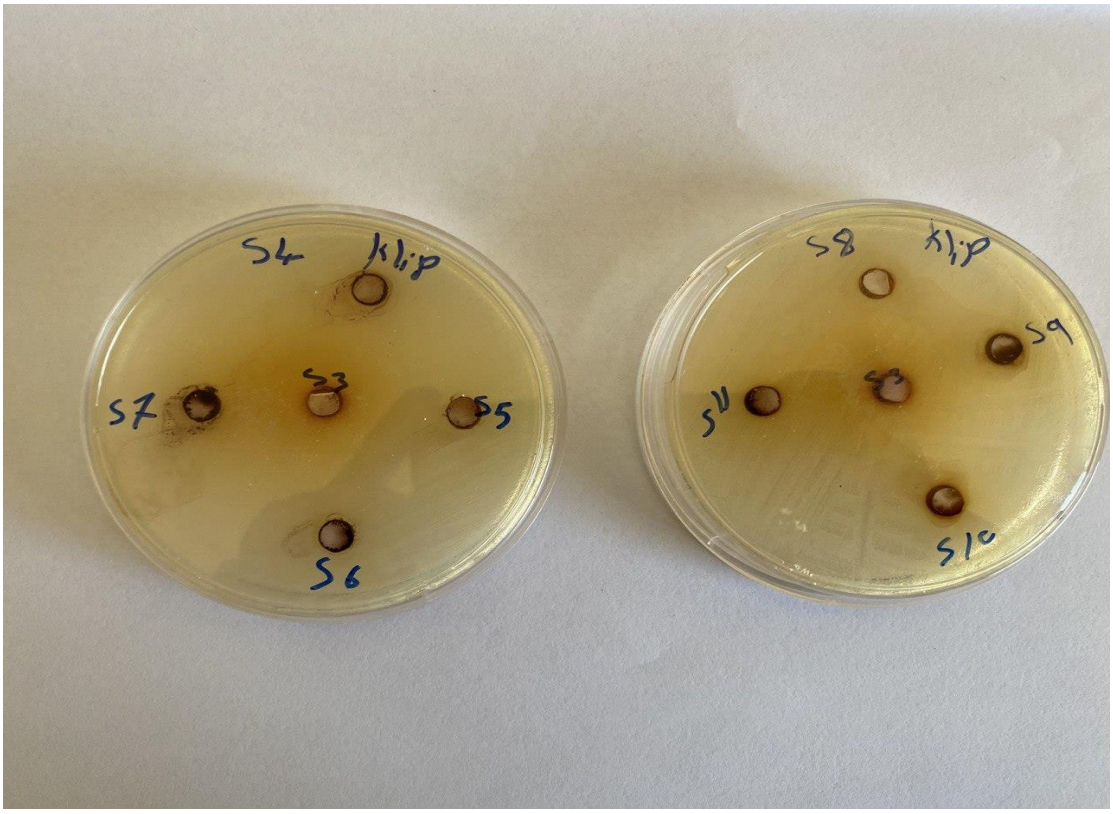


Figure 3-78 : Effect of compounds [S3-S11] on *Staphylococcus epiderimidis , Staphylococcus aureus , Klebsiella pneumoniae ,pseudomonas and E.Coli.*

3.10. Corrosion Measurement :

The Corrosion inhibition of prepared compounds (S12, S13, S15, S18 and S22) have been studied on Carbone Steele surface area in (0.1) M hydrochloric acid solution at different temperature)293, 303, 313 and 323) K. polarization method was used to evaluate the inhibition efficiency of the above compounds, IE%, was calculated in the equation below:

%IE = (Icorr (blank) – (icorr) / Icorr (blank)) × 100

Table (3-4) showed good inhibition efficiency for the synthesis compounds at different temperatures, that give the maximum inhibition efficiency IE %, Results

indicate that ring substitution in the designed compounds has a significant effect on the corrosion inhibition.

Hetero atoms in the compound nucleus, polar functional groups and conjugated double bonds as a part of substitution was indicated to have a significant role on the inhibition efficiency.

Cupper

Table 3-4 : Corrosion parameters for blank and compound in HCl solutions at different temperature range.

Comp.	Temp.	E corr.	I corr.	I corr./ r	Resis.	Anodic β	Cathodic β	Corr. rate,	IE%
Blank	293	0.101-	66.38	6.638E-5	405.4	0.083	0.243	0.326	-
	303	0.122-	70.02	7.002E-5	340.4	0.112	0.108	0.344	-
	313	0.117-	75.24	7.524E-5	544.6	0.111	0.615	0.369	-
	323	0.120-	78.93	7.893E-5	536.4	0.113	0.715	0.387	-
12	293	0.173-	1.999	1.999E-6	3.960E+4	0.396	0.338	0.010	**97**
	303	-0.173	2.424	2.424E-6	3.751E+4	0.474	0.375	0.012	**97**
	313	0.177-	2.765	2.765E-6	3.379E+4	0.494	0.381	0.014	**96**
	323	-0.180	2.853	2.753E-6	3.682E+4	0.558	0.401	0.014	**96**
13	293	-0.160	1.751	1.751E-6	5.874E+4	0.536	0.424	0.009	**97**
	303	-0.163	1.981	1.981E-6	5.570E+4	0.579	0.453	0.010	**97**
	313	-0.164	2.154	2.154E-6	5.495E+4	0.641	0.475	0.011	**97**
	323	0.165-	2.233	2.233E-6	5.192E+4	0.613	0.473	0.011	**97**
15	293	-0.107	11.55	1.155E-5	5293	0.202	0.465	0.057	**83**
	303	-0.066	15.04	1.504E-5	3765	0.171	0.546	0.074	**79**
	313	-0.054	17.48	1.748E-5	3061	0.158	0.562	0.086	**77**

	323	-0.040	19.66	1.966E-5	2649	0.149	0.607	0.097	**75**
18	**293**	-0.194	2.746	2.746E-6	5.021E+4	0.947	0.478	0.013	**96**
	303	-0.195	2.877	2.877E-6	5.071E+4	1.023	0.500	0.014	**96**
	313	0.198-	3.207	3.207E-6	4.065E+4	0.965	0.436	0.016	**96**
	323	-0.192	3.227	3.227E-6	4.687E+4	1.070	0.517	0.016	**96**
22	**293**	-0.051	15.57	1.557E-5	3124	0.149	0.452	0.076	**77**
	303	-0.066	16.25	1.625E-5	3352	0.167	0.504	0.080	**77**
	313	-0.071	18.92	1.892E-5	3227	0.184	0.595	0.093	**75**
	323	-0.047	21.34	2.134E-5	2620	0.166	0.578	0.105	**73**

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

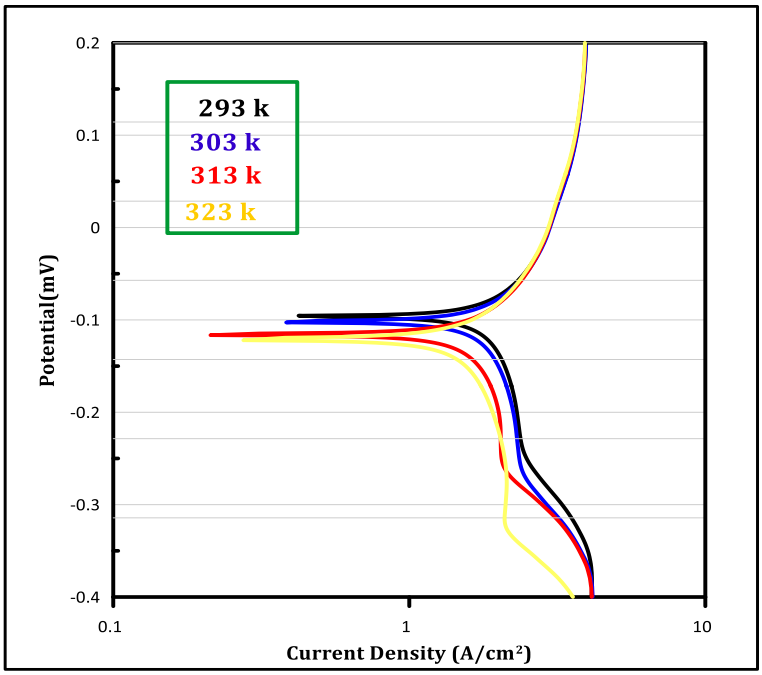


Figure 3-79: Polarization curves for corrosion of blank HCl solution in different temperature.

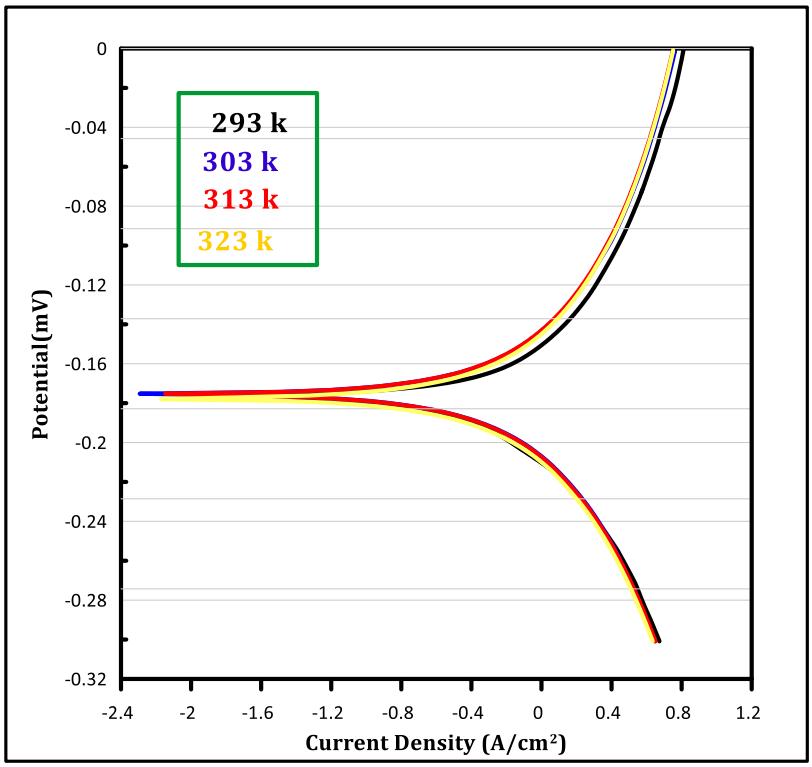


Figure 3-80 : Polarization curves for corrosion of blank HCl solution and S12 in different temperature.

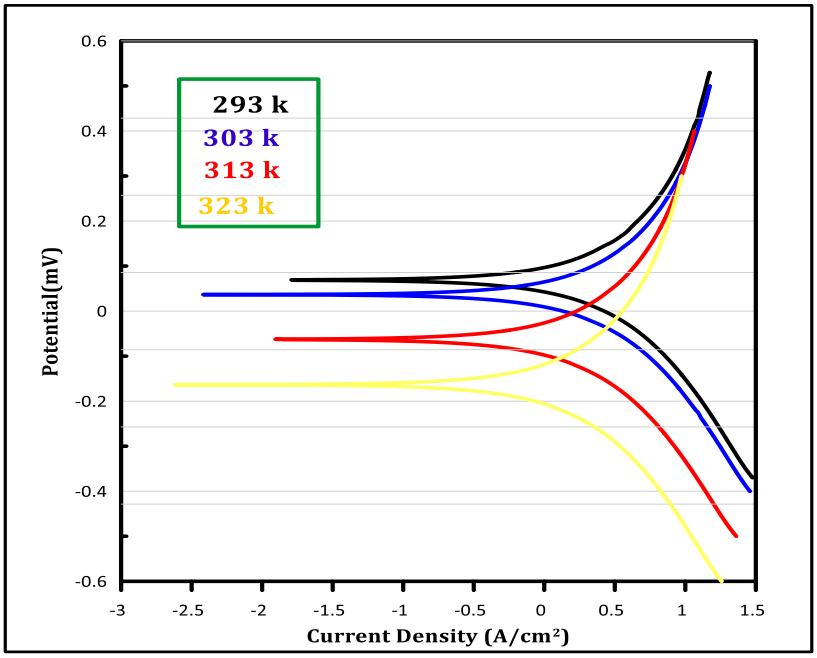


Figure 3-81: Polarization curves for corrosion of blank HCl solution and S13 in different temperature.

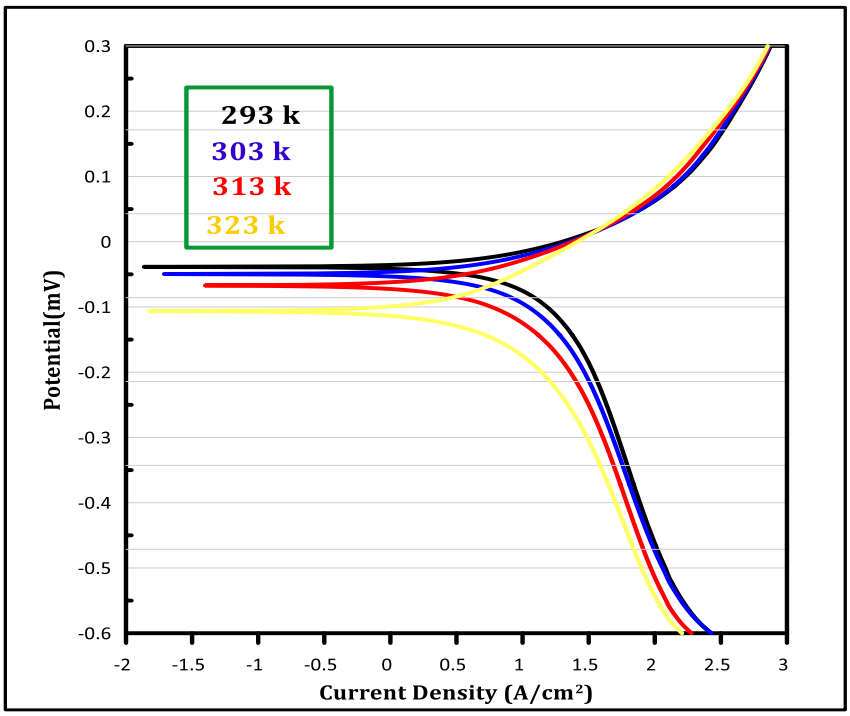


Figure 3- 82:Polarization curves for corrosion of blank HCl solution and S15 compound in different temperature.

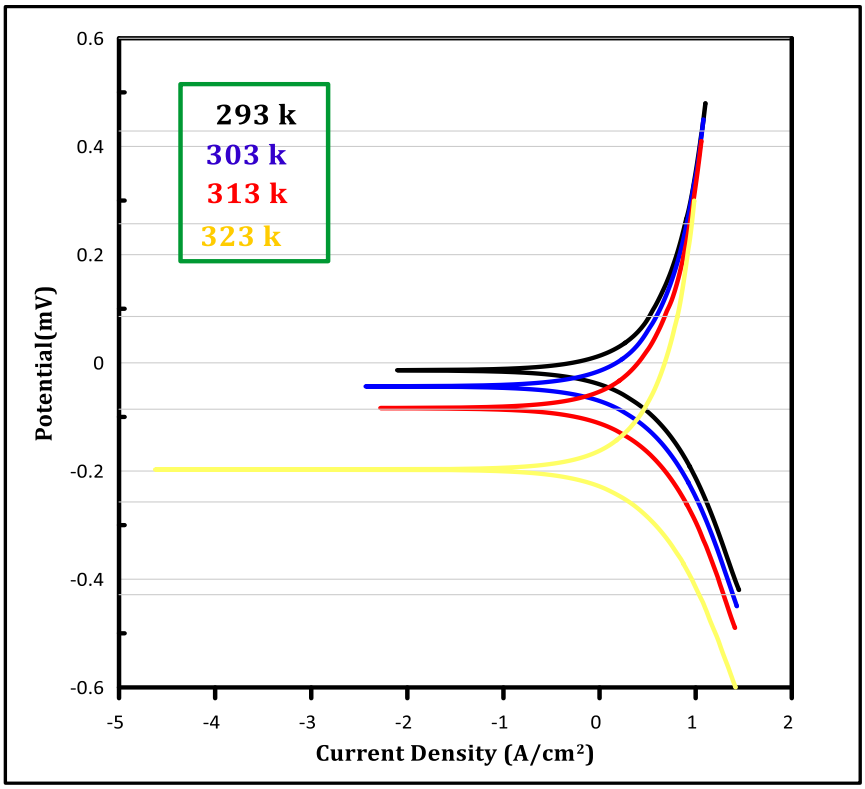


Figure 3-83:Polarization curves for corrosion of blank HCl solution and S18 in different temperature.

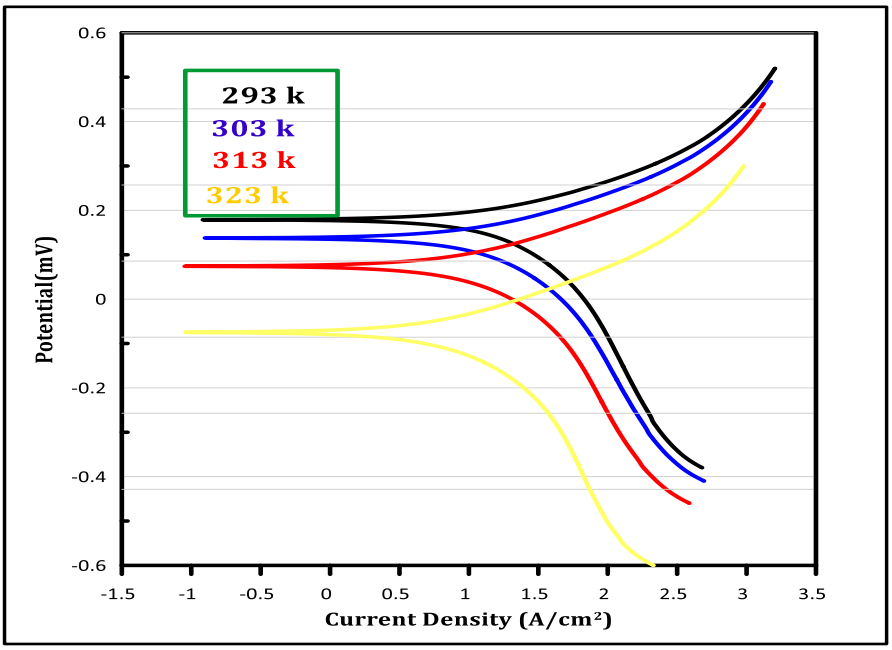


Figure 3 -84: Polarization curves for corrosion of blank HCl solution and S22 in different temperature.

3.11.Conclusions:

1. Synthesis of azo dye ,ester and hydrazide with a high yield .

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

3. Several new Schiff-base heterocyclic compounds were synthesized from 1,3,4-thiadiazole amine with very good yield and confirmed by FTIR, ^{1}HNMR, ^{13}CNMR and CHNS .

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

5.Study the biological activity of the five and six-membered heterocyclic compounds and were achieved excellent results, especially on *pseudomonas* and *E. coli* bacteria . The compounds [S3 and S8] have a good anti-Bacterial activity [30,27] due to their structure that form interaction with wall of cell in selected bacteria which caused high inhibition .

6.Corrosion inhibitor were examined for synthesized compounds (S12, S13, S15, S18 and S22) which exhibited a good inhibition efficiency.

3.12. Suggestions For Future Work :

1. Synthesis of new different azo dyes from the 1,3,4-thiadiazole amine.

2. synthesis of new five heterocyclic compounds like oxadiazole ,triazole, thiazolidine ,thiadiazole derivatives ,imidazole and oxazoline .

3. Synthesis more of new Schiff-base from the 1,3,4-thiadiazole amine.

4. Study the anti cancer for prepared compounds .

5. Study the corrosion for prepared compounds .

6. Synthesis of new six and seven heterocyclic compounds like oxazepine and oxpyridazine

Reference

References :

[1] R. O. C. Norman **,(1974)** principles of organic synthesis John Wiley and Sons, Inc. New York , 313 **.**

[2] .H. L. Yalc and K. Losee,**(1966)** J. *Med. Chem*., 9, 478 .

[3].R.K. Bansal, **(2005)** Heterocyclic Chemistry 4th edition, New Age international (P) Limited, Publishers, New Delhi.

[4]. A. Guzman-Perez, R. T. Webster, M. C. Allen, J. A.Brown, A. R. Buchholz, E. R. Cook, W. W. Day, E. S. Hamanaka, S. P. Kennedy, D. R. Knight, P. J. Kowalczyk, R. B. Marala, C. J. Mularski, W. A. Novomisle, R. B. Ruggeri, W. R. Tracy, R. J. *Hill*, **(2001)** , *Bioorg*, *Med Chem* Lett, 11, 803 **.**

[5] A. E. Amr, N. A. Abdel-Latif, M. M. Abdalla , **(2006)** ,Bioorg *Med Chem* , 14, 373 .

[6] Ali hatem ahmed and Ibtisam K.Jassim , **(2019) ,** Synthesis, identification and antibacterial study for some new heterocyclic compounds ,*Biochem.Cell.Arch*.vol.19,No.2,pp.4563-4571 .

[7] Atia A.J., and Al-Mufrgeiy S.S, **(2012)**,Synthesis and Antibacterial Activities of New 3-Amino-2-Methyl-Quinazolin-4 (3h)-One Derivatives, *Am. J. Chem*;2(3),150-156 **.**

[8]Taj T., Kamble R.R., Gireesh T., and Badami B .V, **(2011)**.An expeditious green synthesis of Schiff bases and azetidinones derivatives with 1,2,4-triazoles, *J. Chem. Sci*,123 (5), 657–666.

[9]. Kahveci, B.; Menteşe, E.; Akkaya, E.; Yılmaz, F.; Doğan, İ.S.; Özel, , **(2014) ,**Synthesis of some novel 1,2,4- triazol-3-one derivatives bearing the salicyl moiety and their anticonvulsant activities, *Arch. Pharm. Chem. Life Sci*., 347, 449-455 **.**

[10] Abdullah H.M., Jassim I.K., Safi M.N., **(2012),**Synthesis and characterization of new heterocyclic compounds with studying its biological activity, *Karbala J. Pharm. Sci*., 4, 115–135 **.**

Reference

[11] Sripriya1 S., Subha C., and Selvaraj A, **(2013),**The Inhibition Chemistry of 2-Amino, 5-Phenyl 1, 3, 4- Triazole for Aluminium in Hydrochloric Acid Solution, *IOSR- JAC*,6 (2), 25-29**.**

[12] Sengupta A.K., and Garg M, **(1988),** Studies on Potential Pestisides-Part XIV Synthesis and Biological Activities of Some New Thiosemicarbazide and Triazole Derivatives, *Def. Sci*., 31 (2): 91-96

[13] Er J.C., Tang M.K., Chia C.G., Liew H., Vendrell M., and Chang Y.T,**(2014) ,** Megastockes BODIPY-triazoles as Environmentally Sensitive Turn-on Fluorescent Dyes, *J. Chem. Sci*.,4: 2168-2176

[14] Bulut VN., Duran C., Gundogdu A., Soylak M, Yildirim N., and Tufekci M., **(2010) ,** A Triazole Derivatives as A New Acid-Base Indicator, Bull. *Chem. Soc. Ethiop*. 24 (3): 457-460 **.**

[15] Cassani S., Kovarich S., Roy PP., VanderWal L., and Gramatica P., **(2013) ,** Daphnia and Fish Toxicity of (Benzo)triazoles: Validated QSAR Models, and Interspecies Quantitative Activity-Activity Modeling,*J.Haz.Mat*.,258-259:50-60 **.**

[16] Nagham M A., **(2016)** ,"Synthesis and Biological Study of Hetero (Atoms and Cycles) Compounds" , Der PharmaChemica, 8(6), 40-48p**.**

[17] Atyaf Y. A. and Nasreen R., **(2016),**Synthesis and Caracterization A New Thiadiazepine Compounds From New Bis 4-Amino-3-Mercpto-1,2,4-Triazol Derivatives, *International Journal Of Research In Pharmacy And Chemistry*; 6(3), 372-378 **.**

[18] Rama D.K., Rao Patnaik K.S.K., Ashok D., Bathula R., Shobha Rani S, Vasudha B., and Gopagoni S., **(2017) ,** Comparison study between convenstional method and mircrowave irradiation method to synthesize oxadiazole derivatives, Int. *J. of Pharmacy and Analytical Research* , 6(2) 396-404**.**

[19] Pagariya R. F. and Thakare N.S., **(2016),** "Synthesis and in vitro antibacterial evaluation of 2, 4- dinitro phenol incorporated azo dyes molecules. "*Der Pharma Chemica*, 8(7):130-136 **.**

Reference

[20] T. Eren; M., Kose; N., Kurtoglu; G., Ceyhan; V. McKee and V. Kurtoglu , **(2015) ,** *Inorg. Chim. Acta*, 430, 268-279.

[21] N.T., Abdel-Ghani; A.M., Mansour Abo El-Ghar; M.F., El-Borady O.M. and Shorafa H , **(2015)** , *Inorg. Chim. Acta*, 435, 187-193.

[22].Ferreira G.R., Marcial B.L., Garcia H.C., Faulstich F. R. L., Dos Santos H. F. and de Oliveira L F.C., **(2015),** *Supramol. Chem.*, 27(1-2), 13-20.

[23] N.S.Al. Obaidi ,O.D .A.sattar ,F.F.Hadi, A.S,Ali,and B.Th.Zaki,

(2018) ,Synthesis and Characterization of Some Azo Dyes Derived from 4-Aminoacetophenone, 1, 4 Phenylene Diamine and Studying its Dyeing Performance and Antibacterial Activity,J *Biochem Tech* 9(4): 33-42.

[24] Pagariya S.K., Pathade R.M. and Bodkhe P.S,**(2015),**Synthesis, Characterization and Antimicrobial screening of some Azo compounds derived from Ethyl vanillin, Res. *J of Chem Sci*, 5(7): 20-28 **.**

[25] Jarad A., **(2012)** , Synthesis and Characterization of New Azo Dye Complexes with Selected Metal Ions, *J of Al- Nahrain University*,15(4): 74-81 **.**

[26] Kate S., and Thakare N.S., **(2016)**, Synthesis and spectral characterisation of some Azo amine Dyes, *journal of Global Biosciences*, 5(1): 3615-3617 **.**

[27]N.A. Alrazzaq ,S.abdulla Aowda and A.Jasim Atiyah , **(2018),** synthesis and characterization of new 1,2,4-triazole derivaties from 2-naphthol ,vol(26).

[28] Abdulridha, A. A. et al. **(2020)** 'Corrosion inhibition of carbon steel in 1 M H2SO4 using new Azo Schiff compound: Electrochemical, gravimetric, adsorption, surface and DFT studies', *Journal of Molecular Liquids*, 315, p. 113690.

[29] B.Manjuntha ,Y.Bodke ,N. Obaiah and Lohith T N, Coumarin-**(2021),** Benzothiazole Based Azo Dyes: Synthesis, Characterization, Computational, Photophysical and Biological Studies .V 1246,131170 *jornal of molecular structure* **.**

[30] J.O.otutui , A.K. asiagwui, and P.O. itoya , **(2019) ,**disperse dyes based on 2-amino-5-mercapto-1,3,4-thiadiazole and 2-amino-4,6 dihydroxypyrimidine moleties ;synthesis and application on nylon 6 fabrics,*Nig.J.Pure and Appl.Sci.*Vol.32**.**

Reference

[31]C.Gopi ,V.G Sastry and M.D. Dhanaraju , **(2017),**synthesis, spectroscopic characterization, X-ray crystallography structural activity relationship and antimicrobial activity of some novel 4-(5-(10-(3-N, N dimethylamino)propyl)-10H-phenothiazine yl)-1, 3, 4-thiadiazole-2-yl) Azo dye/Schiff base derivatives ,v (3),79-89 **.**

[32] V. A. Sawarkar **,(2015) ,** *International Journal of Scientific and Research Publications*, 5, 1 .

[33] K. Nageswarano, K. Venkateswararao and G. Prasad , **(2017) ,** *Journal of Chemical and Pharmaceutical Research* **,** 9 (4), 256 .

[34] M .M.Kareem ,N.A .Alrazzaq , S.A.Aowda and .n.m.Aljamali , **(2021)** ,Synthesis, Characterization and Biological activity study for new hybrid polymers by grafting 1,3,4-triazole and 1,2,4-oxadiazle moieties onto polyvinyl chloride ,*Egypt.J.Chem* .Vol .64.No.3pp.1273-1283**.**

[35] M. F. Mahdi, N. H. Naser and N. H. Hammud, **(2017),** *International*

Journal of Pharmacy and Pharmaceutical Science; 9, 7.

[36] Asif M and Singh A **,(2016),** AnticonvulsantActivitiesof4-benzylidene- 6-(4-methyl-phenyl)-4, 5-dihydropyridazin-(2H)-ones and 4- benzylidene-6-(4-chloro-phenyl)-4, 5-dihydropyridazin-(2H)- ones. *Open Pharmaceutical Sciences Journal* 3.

[37] A. Kumar, B. R. Parveen and Vaishali :**(2017)** *International Journal* of *Research in Pharmacy* and Chemistry ,7 (1), 85 .

[38] M. Faizi, R.Jahani, S. A. Ebadi, S. A. Tabatabai, E. Rezaee, M. Lotfaliei, M. Amimi and A. Almasirad; **(2017)** , *Excil Jouranal*, 16, 52.

[39] A.A.Saheeb and W.K.Damdoom, **(2020)** ,Synthesis ,characterization of new Hydrazone derivatives containing heterocyclic meioty ,vol.23 (16).

[40] H.H. Sallama, Y. H.I.Mohammed, F.H. Al-Ostootc,e, S.M.A., and S.A. Khanum , **(2021)** ,Synthesis, crystal structure characterization, DFT calculations,

Reference

Hirshfeld surface analysis and 3D energy frameworks of triazole pyridazine derivatives: Theoretical and experimental studies,

Journal of Molecular Structure 1246 **,**131242.

[41] N.A.Alrazzaq , **(2007)** synthesis of New Heterocyclic Compounds derived from m-methyl benzoic acid and P-nitro benzoic acid and study of Biological activity for some of these compounds **.**

[42] A.O.Abdlhamid, I E. El Sayed, Y.H. Zaki, A.M. Hussein, M. M. Mangoud and M. A. Hosny , **(2019),** Utility of 5-(furan-2-yl)-3-(p-tolyl)-4,5-dihydro-1-H-pyrazole-1-carbothioamide in the synthesis of heterocyclic compounds with anti microbialactivity ,*BMC Chemistry* 13:48 **.**

[43] A.H.Samir , Synthesis, Characterization and Antimicrobial Activity of New 2-Phenylquinoline-4(3H)-one Derivatives , **(2017),** *Orient.J.Chem.*,Vol.33(3),1422-1432 **.**

[44] A.A.Majeid ,A.A .Aly ,M.S.Amine and A.A.Ghith , **(2016).** Synthesis and Investigation of Mass Spectra and Antimicrobial Activity of Some Azoles and Azines Based on (quinolone-8-yloxy) acetohydrazide ,*Int.J.Nano.Chem* .2,37-45 **.**

[45] Renuka, N.; Kariyappa, A. K. **(2013)** Synthesis and biological evaluation of novel formyl-pyrazoles bearing coumarin moiety as potent antimicro- bial and antioxidant agents. *Bioorg. Med. Chem. Lett***,** 23, 6406− 6409.

[46]Kalaria, P. N.; Shailesh, P. S. Dipak, K. R. **(2014),** Synthesis, identification and in vitro biological evaluation of some novel 5-imidazopyrazole incorporated pyrazoline and isoxazoline derivatives. *New J. Chem.* **,** 38, 2902−3002.

[47] a) El-Metwaly, N.; Katouah, H.; Aljuhani, E.; Alharbi, A.; Alkhatib, F.; Aljohani, M.; Alzahrani, S.; Alfaifi, M. Y.; Khedr, A. M. **(2020)** Synthesis and elucidation for new nanosized Cr (III)-pyrazolin complexes; crystal surface properties, antitumor simulation studies beside practical apoptotic path. J. *Inorg. Organomet. Polym* **,** 30, 4142−4154. (b) El-Metwaly, N.; Althagafi, I.; Khedr, A. M.; Al-Fahemi, J. H.; Katouah, H. A.; Hossan, A. S.; Al-Dawood, A. Y.; Al-Hazmi, G. A **(2019)**, complexes-dyes and their usage in dyeing cotton to be special bandage

for cancerous wounds. *J. Mol. Struct*. 1194, 86−103. (c) Althagafi, I.; El-Metwaly, N. M.; Farghaly, T. Characterization of new Pt (IV)− thiazole complexes: Analytical, spectral, molecular modeling and molecular docking studies and applications in two opposing pathways. Appl. Organomet. Chem**.** 33, No. e5099.

[48] Yang, J.; Gharagozloo, P.; Yao, J.; Ilyin, V. I.; Carter, R. B.; Nguyen, P.; Robledo, S.; Woodward, R. M.; Hogenkamp , D. J. **(2004),** 3-Phenoxyphenyl pyrazoles: a novel class of sodium channel blockers. *J. Med. Chem*. 47, 1547−1552

[49] El-Metwaly, N.; Farghaly, T. A.; Elghalban, M. G.**(2020),** Synthesis, analytical and spectral characterization for new VO (II)-triazole complexes; conformational study beside MOE docking simulation features. *Appl. Organomet. Chem* 34, No. e5505.

[50] Dale D J, Dunn P J, Golightly C, Hughes M L, Levett P C,PearceAK,SearlePM,WardG and WoodAS **(2000)** , The chemical development of the commercial route to sildenafil: a case history The chemical development of the commercial route to sildenafil: A case history Org. Process. *Res. Dev*. 4 17

[51] DaiH,ChenJ,LiG,GeS,ShiY,FangYandLingY, **(2017),** Design, synthesis, and bioactivities of novel oxadiazole-substituted pyrazole oximes *Bioorg. Med. Chem*. Lett. 27 950

[52] Alam R, Wahi D, Singh R, Sinha D, Tandon V, Grover A and Rahisuddin Design, **(2016)** synthesis, cytotoxicity, HuTopoIIa inhibitory activity and molecular docking studies of pyrazole derivatives as potential anticancer agents *Bioorg. Chem*. 69 77

[53] Chougala B M, Samundeeswari S, Holiyachi M Shastri L A, Dodamani S, Jalalpure S, Dixit S R, Joshi S D and Sunagar V A**(2017)** ,Synthesis, characterization and molecular docking studies of substituted 4-coumarinylpyrano[2,3-c]pyrazole derivatives as potent antibacterial and anti-inflammatory agents Eur. *J. Med Chem*. 125 101

[54] S.J.Mohammed,F.G.Zuhier ,B.F.Salou, **(2018)** Synthesis and Characterization of Some Pyrazole , Pyrazoline and Pyrazolidine Derivatives ,vol.xi,issue 1,issn 2277-8322 **.**

Reference

[55] M Amir and S Kumar**,(2005),**Synthesis and anti-inflammatory, analgesic, ulcerogenic and lipid peroxidation activities of 3,5-dimethyl pyrazoles, 3-methyl pyrazol-5-ones and 3,5-disubstituted pyrazolines, *Indian Journal of Chemistry,*Vol. 44B, pp. 2532-2537

[56] E.M. Flefel , W. I. El-Sofany , M. El-Shahat , A. Naqvi and E. Assirey, **(2018)** , Synthesis, Molecular Docking and In Vitro Screening of Some Newly Synthesized Triazolopyridine, Pyridotriazine and Pyridine–Pyrazole Hybrid Derivatives ,*Molecules* **,**23,2548

[57] A.Kudelko,M. Olesiejuk, M.Luczynski, M.Swiatkowski, T.Sieranski and R.Kruszynski, **(2020)** 1,3,4-Thiadiazole-Containing Azo Dyes: Synthesis, Spectroscopic Properties and Molecular structure ,*Molecules* ,25,2822 .

[58] Abdel-Wahab, B.F., Abdel-Aziz, H.A., Ahmed, E.M. **(2009)** Synthesis and antimicrobial evaluation of some 1,3-thiazole, 1,3,4-thiadiazole, 1,2,4-triazole, and 1,2,4-triazolo[3,4-b] [1,3,4]-thiadiazine derivatives including a 5-(benzofuran-2-yl)-1-phenylpyrazole moiety. *Monatsh Chem*, 140: 601–605.

[59]Matysiak, J., Malinski, Z. **(2007)** 2-(2,4-dihydroxyphenyl)-1,3,4 thiadiazole analogues: antifungal activity In Vitro against Candida species. Rus *J Bioorg Chem* ; 33: 594-601.

[60] Kadi, A.A., Al-Abdullah, E.S., Shehata, I.A., Habib, E.A., Ibrahim, T.M., El-Emam, A.A. **(2010)** Synthesis, antimicrobial and anti-inflammatory activities of novel 5-(1-adamantyl)-1,3,4- thiadiazole derivatives. *Euro J Med Chem* **,**45: 5006-5011.

[61] Zheng, K.B., He, J., Zhang, J. **(2008)** Synthesis and antitumor activity of N1-acetylamino- (5- alkyl/aryl-1,3,4-thiadiazole-2-yl)-5-fluorouracil derivatives Chin *Chem Lett*, 19: 1281-1284.

[62] Hamad, N.S., Al-Haidery, N.H., Al-Masoudi, I.A., Sabri, M., Sabri, L., Al-Masoudi, N.A. **(2010);** Amino acid derivatives, part 4: Synthesis and anti-HIV activity of new naphthalene derivatives. *Arch Pharm Chem Life Sci***,**343: 397-403.

Reference

[63] Chen, Z., Xu, W., Liu, K., Yang, S., Fan, H., Bhadury, P.S., Hu, D., Zhang, Y. **(2010)** , Synthesis and antiviral activity of 5-(4-chlorophenyl)-1,3,4-thiadiazole sulfonamides. Molecules,; 15: 9046-9056 .

[64] C.T.k.Kumar,J.Keshavayya,T.N.Rajesh,S.K.Peethambar ,and A.R.Sh.Ali , **(2013)** Synthesis, Characterization, and Biological activity of 5-Phenyl-1,3,4-thiadiazole-2-amine Incorporated Azo Dye Derivatives,370626 **.**

[65] S.Adnan , A. J. Mohammed and H. Thamer , **(2015),**Synthesis and identification of some derivatives of 1,3,4-thiadiazole,*J.Chem.pharm*.res,7,(10):1000-1011 .

[66] N.M. Aljamali, N .S. M .Husien , Dr .F.A. Wannas and Dr.A.B.Aldujaili , **(2020)** Review on Preparation and Applications of Thiadiazole Derivatives ,vol .6:issue 1 .issn:2582-5917.

[67] T.S.Kokovina ,S.Y.Gadomsky ,A.A.Terentiev and N.A.Sanina , **(2021)** ,A Novel Approach to the Synthesis of 1,3,4-Thiadiazole-2-amine Derivatives ,molecules **,** 26,5159 .

[68] Shi, M. et al. **(2019)** 'Tuning of reversible thermochromic properties of salicylaldehyde Schiff bases through the substitution of methoxy and nitro groups', *Journal of Molecular Structure*, 1182, pp. 72–78. doi: 10.1016/j.molstruc.01.012

[69] M. M. Abo-Aly, A. M. Salem, A. M. Sayed and A. A. A. Aziz

(2015) Molecular and Biomolecular Spectroscopy**,** 136, 993.

[70] A. A. A. Aziz, A. N. M. Salaem, M. A. Sayed and M. M. Aboal.

(2015), *Journal of Molecular structure***,**1010, 130.

[71] A. A. M. Belal, I. M. El-Deen, N. Y, Farid, Z. Rosan and S. R

Moamen; **(2015)** *Journal of Molecular structure*, 149, 771.

[72] C. U. Dueke- Eze, T. M. Fasina and N. Idika; **(2011)** African *Journal of Pure and applied Chemistry***,** *5, 2.*

Reference

[73] A .Kajal , S .Bala, S .kamboj ,N .sharma and V .Saini, **(2013),** Schiff base : A versatile pharmacophore , *journal of catalyst* **.**

[74] R.Kais and S. Adnan , Synthesis, **(2019)** Identification and Studying Biological Activity of Some Heterocyclic Derivatives from 3, 5-Dinitrosalicylic acid,IOP Conf. Series: *Journal of Physics: Conf. Series* 1234 **,**012091 .

[75] Pagariya S.K., Pathade R.M. and Bodkhe P.S. **(2015),**Synthesis, Characterization and Antimicrobial screening of some Azo compounds derived from Ethyl vanillin, *Res. J of Chem Sci,* 5(7): 20-28 **.**

[76] Jarad A, **(2012)** Synthesis and Characterization of New Azo Dye Complexes with Selected Metal Ions, J of Al- Nahrain University,15(4): 74-81 **.**

[77] Kate S., and Thakare N.S., **(2016),**Synthesis and spectral characterisation of some Azo amine Dyes, journal of Global Biosciences, 5(1): 3615-3617 **.**

[78] S.A.Karim ,H.Y Al.Gubury and N.A.Alrazzak, **(2019)** the Synthesis of a Novel Azo Dyes and Study of Photocatalytic Degradation, Conf. Series 1294 .

[79] Asif, M. Chron **(2010),** Young Sci **,** 1, 3.

[80] Asif, M.; Singh, A.; Ratnakar, L. **(2011)** J Pharm Res, 4, 664.

[81] Asif, M. **(2010)** *J Adv Sci Res***,** 1, 35.

[82] Asif, M ; Singh, A; Siddiqui, A. A. **(2012),** *Med Chem Res* **,** 2,3336.

[83] Babu, P. A; Narasu, M. L.; Srinivas, K. ARKIVOC **,** 247.

[84] DaiH,ChenJ,LiG,GeS,ShiY,FangYandLingY **(2017)** Design, synthesis, and bioactivities of novel oxadiazole-substituted pyrazole oximes *Bioorg*. *Med. Chem. Lett*. 27 950.

[85]Alam R, Wahi D, Singh R, Sinha D, Tandon V, Grover A and Rahisuddin**(2016)** Design, synthesis, cytotoxicity, HuTopoIIa inhibitory activity and molecular docking studies of pyrazole derivatives as potential anticancer agents *Bioorg. Chem*. 69 77.

Reference

[86] Chougala B M, Samundeeswari S, Holiyachi M,Shastri L A, Dodamani S, Jalalpure S, Dixit S R, JoshiS D and Sunagar V A **(2017)** Synthesis, characterizationand molecular docking studies of substituted 4-coumarinylpyrano[2,3-c]pyrazole derivatives as potent antibacterial and anti-inflammatory agents Eur. *J. Med.Chem.* 125 101.

[87] M .Konwara, H. M .F .Elnagdya, P.S.Gehlotb, N. D khupse , A. Kumarb ,and D.Sarma, **,(2019)**Transition metal containing ionic liquid-assisted one-pot synthesis of pyrazoles at room temperature,*J.Chem.Sci***.**131:80.

[88] P. Maaß, **(2011),**Corrosion And Corrosion Protection, Wiley-Vch Verlag GmbH and Co. KGaA,.

[89]G.V. Redkina, A.S. Sergienko and Yu.I. Kuznetsov, **(2020)** Hydrophobic and anticorrosion properties of thin phosphonate–siloxane films formed on a laser textured zinc surface, *Int. J. Corros. Scale Inhib.*, 9, no. 4, 1550–1563.

[90] A.I.Biryukov, O.A.Kozaderov, R.G.Galin, D.A.Zakharyevich and V.E.Zhivulin, **(2020),**Details of the mechanism of dissolution of iron–zinc coatings based on the δ-phase in acidic media, *Int. J. Corros. Scale Inhib*., 9, no.4, 1477–1489.

[91] P.P.Kumari, P. Shetty, S.A.Raol, D. Sunil and T Vishwanath, **(2020)** Synthesis, characterization and anticorrosion behaviour of a novel hydrazide derivative on mild steel in hydrochloric acid medium, Bull. *Mater. Sci*. 43:46 .

[92]DuttaA,SahaSK, BanerjeeP and SukulD ,**(2015),** *Corros.Sci*.98 541

[93] Yilmaz N , Fitoz A, Ergun Y and Emregül K C**(2016)** *Corros*.

Sci. 111 110.

[94] Nour Abd Alrazzak , **(2018)**, Synthesis of some New Heterocyclic Derivatives and some of their composites with PVC and Study their PhotoCatalytic Behaviour.

[95] J. Anderson and Dovid, **(1990),**C. A., Vol. 112, 178982r

[96] A. S. Shubrem, **(2006).**M. Sc. Thesis College of Science, Al-Nahrain University, Iraq **.**

الخلاصة

تتضمن هذه الدراسة تخليق مركبات حلقية جديدة غير متجانسة جديدة باستخدام حمض بارا أمينوبنزويك وريسورسينول كمواد أولية لإنتاج الجزيئات الوسيطة والجزيئات المستهدفة. تتضمن هذه الدراسة أيضًا تخليق حلقات مركبة حلقية غير متجانسة مكونة من خمسة وستة أعضاء جديدة [S4-S11] بدءًا من هيدرازيد الحمض [S3] وتوليف المركبات الحلقية غير المتجانسة الجديدة المختلفة قواعد شف [S24-S29]من 4،3،1-ثياديازول أمين [S12]. تم تمييز المواد العضوية باستخدام تقنيات مختلفة: طيف الاشعة تحت الحمراء (FT-IR)، طيف الرنين النووي البروتوني (^{1}H-NMR)، طيف الرنين النووي الكاربوني (^{13}C-NMR) و التحليل النوعي للعناصر (CHNS).

ينقسم هذا العمل إلى خمسة أجزاء مختلفة :

الجزء الاول :

يتضمن هذا الجزء تخليق صبغة الآزو عن طريق التفاعل بين حمض بارا أمينوبنزويك وريسورسينول ، مركب الازو [S1] تم تحويله إلى مركب استر [S2] يتفاعل مع هيدرازين هيدرات لإعطاء مشتق الهيدرازيد. وتحضير مشتقات البيريدازين -6،3-ديون والفثالازين [S4-S7] من حمض هيدرازيد [S3] كما هو موضح في المخطط1.

الجزء الثاني :

تضمن هذا الجزء تخليق مشتقات البيرازول [S8-S11] من تفاعل حمض هيدرازيد [S3] مع (أسيتيل أسيتون ، إيثيل أسيتو أسيتات ، ميثيل أسيتو أسيتات ، ثنائي إيثيل مالونات) في الإيثانول المطلق كما هو موضح في المخطط 2.

الجزء الثالث:

تضمن هذا الجزء تخليق 4،3،1-ثياديازول أمين [S12] عن طريق تفاعل صبغة آزو [S1] مع ثيوسيميكاربازيد وأصباغ آزو الجديدة [S13-S23] من تفاعل ثياديازول [S12] مع (أورسينول ، هيدروكينون ، ريسورسينول ، كاتيكول ، الفا نفثول ، بيتا نفثول ،2-مثيل نفثول ،6,2- ثنائي مثيل فينول ,4,2,1-ترايزول , 4,2,1-ثايول ترايزول ، بارا توليدين)كما هو موضح في المخطط 3.

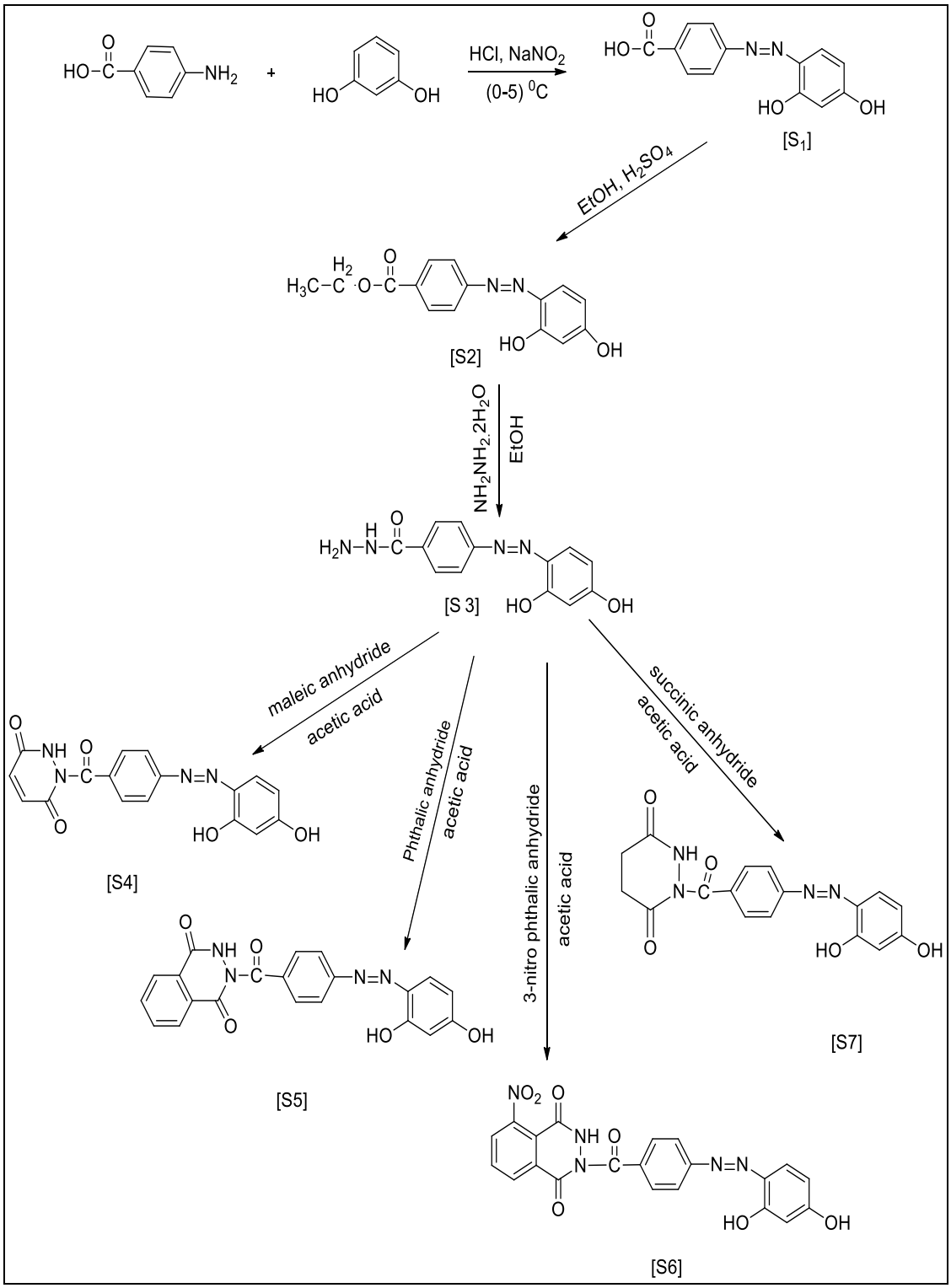
الجزء الرابع:

تضمن هذا الجزء تخليق قواعد شف جديدة [S24-S29] من تفاعل 4،3،1- ثياديازول أمين [S12] مع الألديهيدات الأروماتية (9-أنثرالديهيد ، تريفثالديهيد ، N، N ثنائي ميثيل أمينو بنزالديهايد ، 5،3-ثنائي كلورو ساليسالدهيد ، 3- hydroxybenzaldehdye، 2-hydroxy-5-nitro benzal dehyde) في الإيثانول المطلق كما هو موضح في المخطط 4.

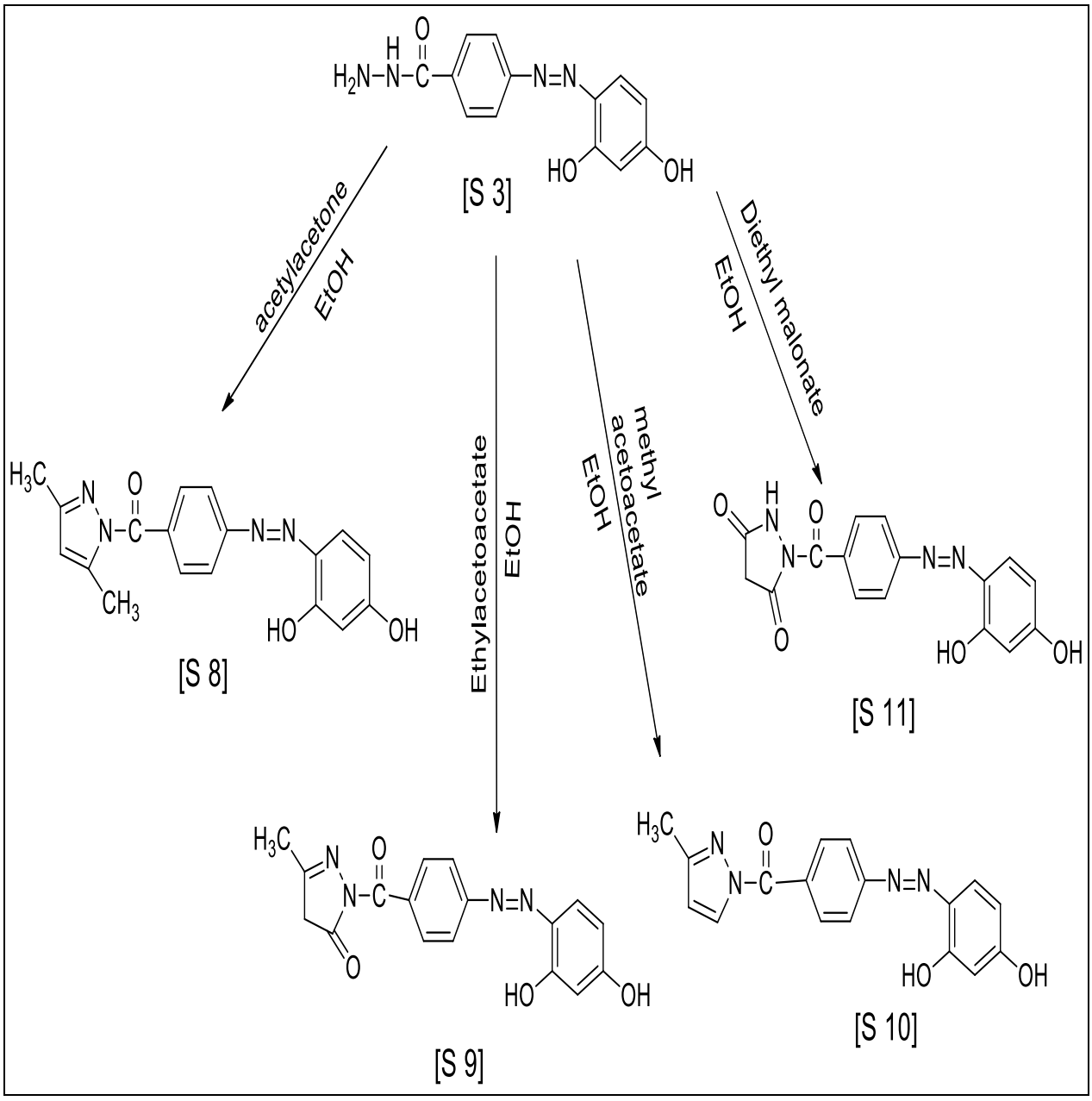
الجزء الخامس :

تضمن هذا الجزء دراسة الأنشطة المضادة للبكتيريا لبعض المركبات المحضرة وتم اختبار هذه الأنشطة بطريقة قرص الأجار ضد خمس سلالات ممرضة من البكتيريا (*Staphylococcus epiderimidis* و *Staphylococcus aureus* و *Klips* و *pseudomonas* و *E.Coli*) كما هو موضح في الجدول3-1.

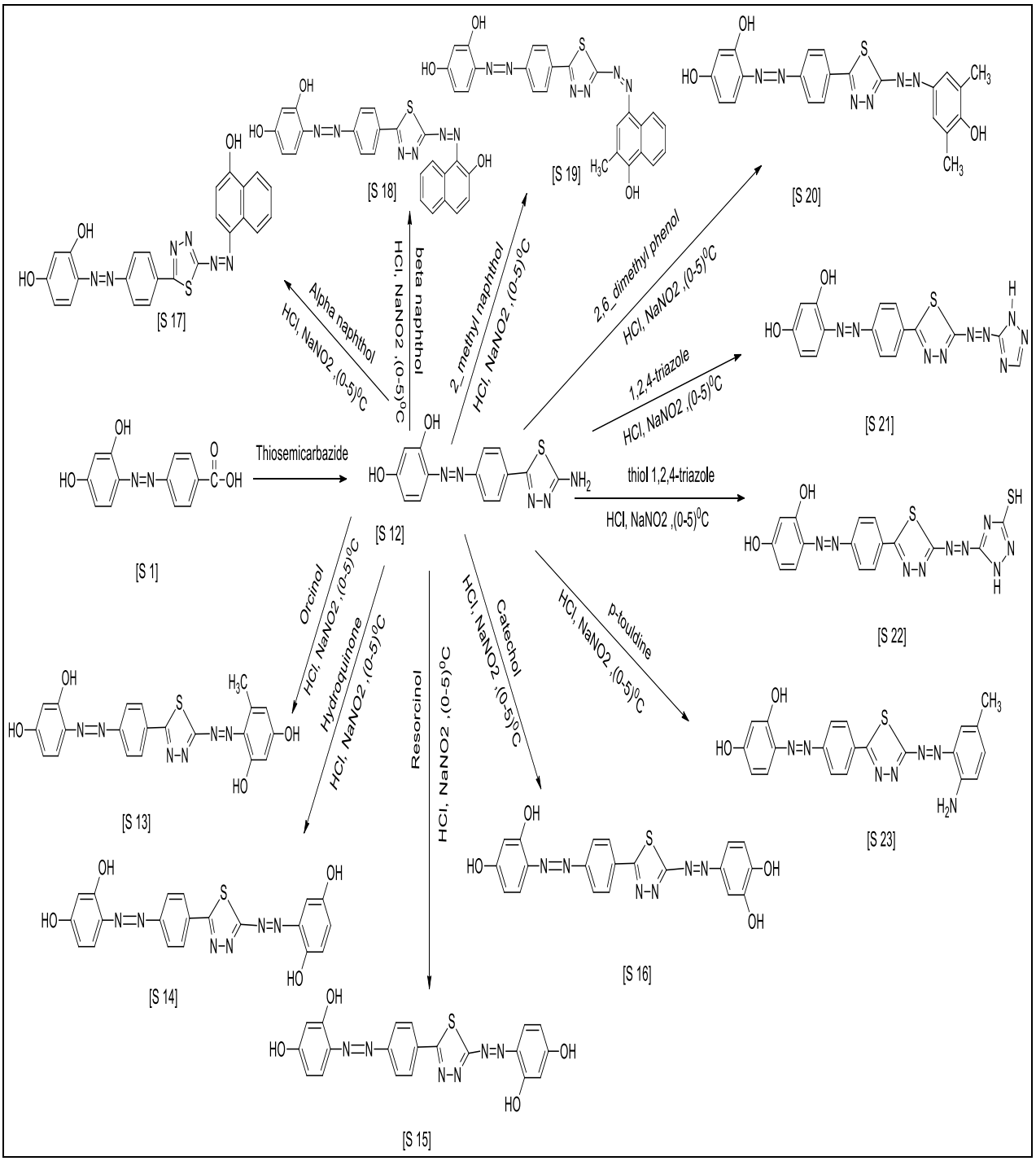
وأيضا تم دراسة مقاومة التآكل لبعض المركبات المحضرة.



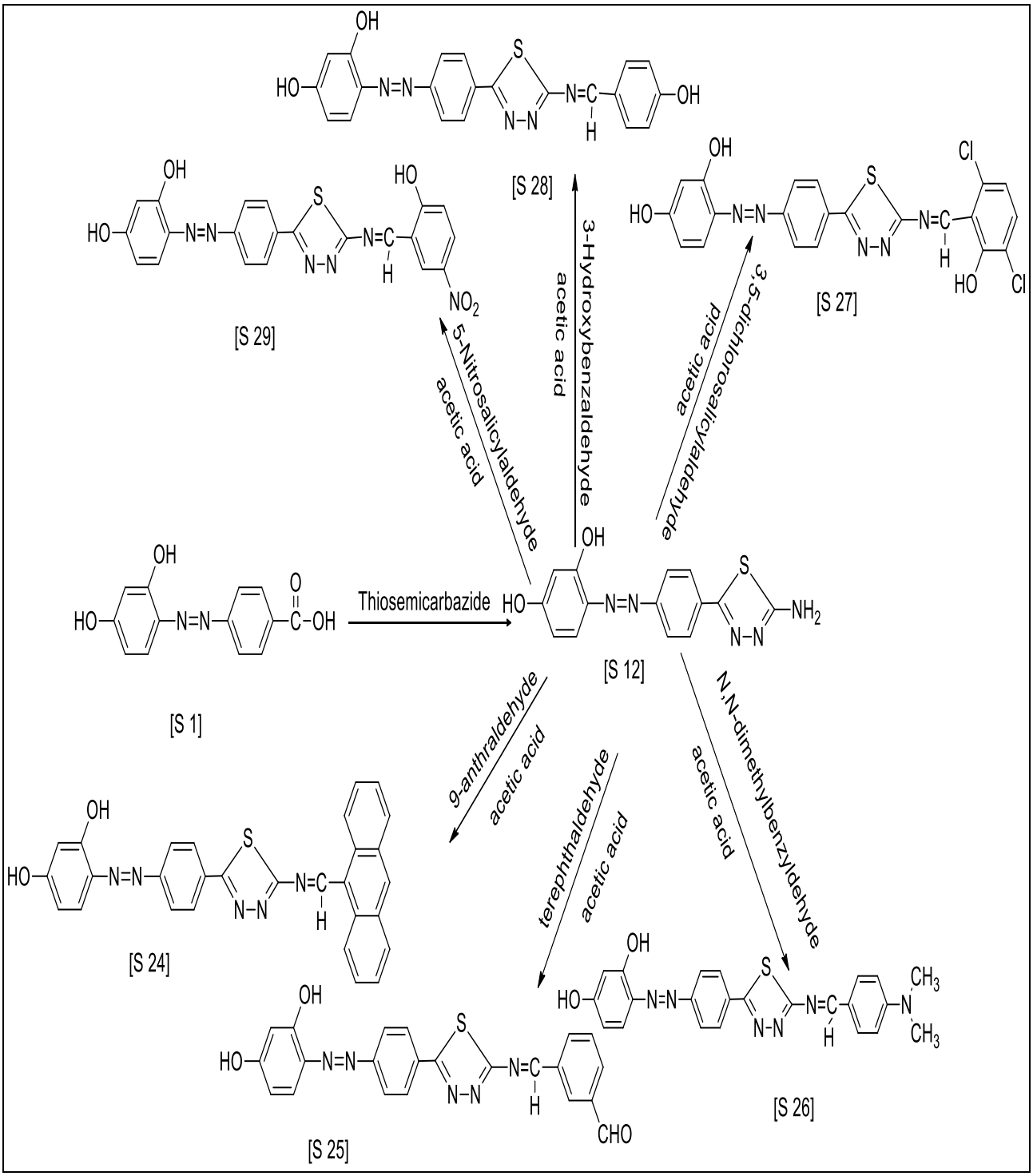
مخطط 1: تحضير المركبات [S1-S7]



مخطط 2: تحضير المركبات [S8-S11]



مخطط 3: تحضير المركبات [S12-S23]



مخطط 4: تحضير المركبات [S24-S29]

جمهورية العراق
وزارة التعليم العالي و البحث العلمي
جامعه بابل
كلية العلوم للبنات
قسم الكيمياء

تحضير و تشخيص المركبات الحلقية غير المتجانسة الجديدة وتطبيقاتها

رسالة

مقدمة الى مجلس كلية العلوم للبنات- جامعة بابل

كتنفيذ جزئي لمتطلبات الحصول على درجة ماجستير العلوم في الكيمياء

شهد محمد جاسم الصافي

بكالوريوس جامعة كربلاء 2019

اشراف

أ. م. د. نور عبد الرزاق عبد اللطيف

1444 هـ

2022 م