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# Preparation, spectroscopic and biological study of some derivatives of formazan and new heterocyclic compounds

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**Abstract**---In this study, a group of Schiff base derivatives and a group of formazan derivatives were prepared , reacted 2-amino-6-methoxy benzothiazole with 3-hydroxy-4-methoxy benzaldehyde to from the derivative of Schiff ,s Base then reactance the derivative of Schiff ,s Base  $\text{NaN}_3$  to from Tetrazole derivative (30) , as well as the reaction of the Schiff derivative 2-amino thiazole to from formazan derivative (31) , and the reaction of the Schiff derivative with 3-amino pyridine to from the formazan derivative (32) .

**Keywords**---Schiff base,  $\text{NaN}_3$ , 2-amino thiazole, 3-amin pyridine.

## Introduction

The Azo Compounds are Compounds that contain the functional group  $\text{R-N=N-R}_2$  Where R ,  $\text{R}_2$  , It could be a group Aryl or Alkyl and functional group is called the azo group , It was discovered and named in 1860 by scientist Greiss<sup>(1)</sup> , These two atoms act a bridge group associated with many different groups , whether aliphatic or aromatic , Aliphatic azo dyes are less common due to their rapid decomposition into nitrogen and hydrocarbons. If they are associated with aromatic groups , they are called aromatic azo dyes <sup>(2)</sup> . It has gained wide popularity due to its high stability , The reason for the stability of the aromatic azo compounds is due to the fact that they contain the azo group ( $-\text{N}=\text{N}-$ ) . With a strong double bond and the number of groups linked on both sides of the azo group<sup>(3,4)</sup> . Many heterocyclic compounds can be biosynthesized by plants or animals . The human body is able to manufacture the oxygen-carrying

hemoglobin pigment in the blood <sup>(5)</sup>. As well as anti-inflammatory allergy and anti-HIV<sup>(6)</sup>.

### **Material**

FT-IR Spectrum ( $400\text{--}4000\text{ cm}^{-1}$ ) in KBr desk recorder on SHIMADZU, made in Thi Qar University, Iraq. Melting Point were measured using Stuart, made in Al Qadisiyah University, Iraq.

### **Method**

#### **Synthesis the Tetrazole derivatives (30)**

Dissolve (0.00095) mole (0.3) gram from the derivative of the Schiff base in (30) ml from 1,4 Dioxane in a circular flask with magnetic stirrer and condenser, Then mix it with (0.00095) mole (0.0617) gram from sodium azide, The mixture was heated for (65 hrs) and temperature ( $55\text{ }^{\circ}\text{C}$ ), The reaction was followed by thin layer chromatography TLC, Using the mobile phase (hexane-ethanol) and Rate (2:8), Then the precipitate was filtered, dried, and recrystallized with absolute ethanol.

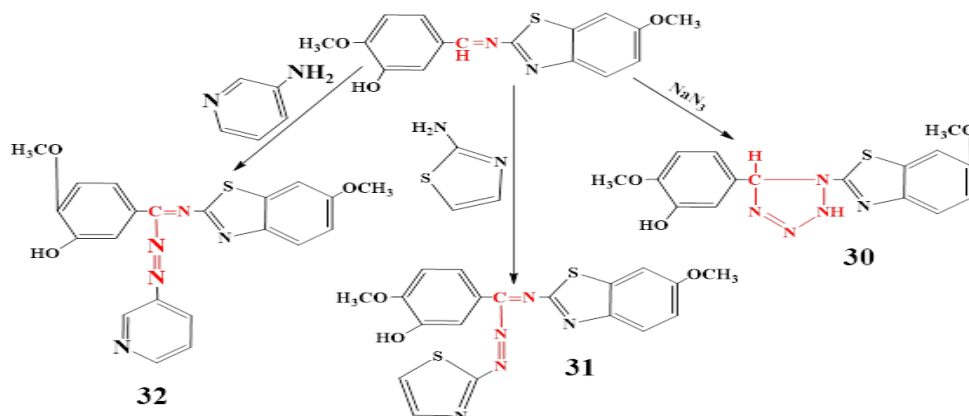
#### **Synthesis the formazan derivatives (31, 32)**

Dissolve (0.00127) mole (0.127) gram from (2-amino thiazole, 3-amino pyridine) in (4 ml HCl + 30 ml distilled water) The mixture was cooled to a temperature of ( $0\text{--}5\text{ }^{\circ}\text{C}$ ), In an ice bath, then add all the second mixture consisting of melting (0.00127) mole (0.088) gram from  $\text{NaNO}_2$  in (10) ml from distilled water Coolant temperature range ( $0\text{--}5\text{ }^{\circ}\text{C}$ ), Drop by drop to the first solution with constant stirring for (20-30) minute. Then all the solution formed from the first and second mixture was added to the third mixture formed of melting (0.00127) mole (0.4) gram from Schiff base and (1) gram from NaOH Solute in (30) ml from distilled water and (5) ml from absolute ethanol, The coolant is in ice bath at a temperature of ( $0\text{--}5\text{ }^{\circ}\text{C}$ ) Leave the solution with continuous stirring for (15-30) minute. Then the solution was neutralized to  $\text{pH} = 6$  with solution NaOH, Then leave the solution while stirring (30-60) minute, Then the precipitate was filtered and washed with distilled water from (3-5) times, It was dried and recrystallized with absolute ethanol.

#### **Preparation Medium Microbiology**

10 g of a gar was dissolved in (300 ml) of distilled water and then placed in an Autoclave for 30 minutes at  $180\text{ }^{\circ}\text{C}$  for Sterilization After reaching  $38\text{ }^{\circ}\text{C}$ , the media are poured into petri dishes, ready for Slicing with bacteria. Staphylococcus aureus and Escherichia Coli were obtained in hospital isolation. These plates were cultured and incubated in  $37\text{ }^{\circ}\text{C}$  for 24 hr Both types of bacteria.

**Scheme : Synthesis the derivatives ( 30 , 31 , 32 )**



**Compound (30) : 2-methoxy-5-[1-(6-methoxy benzothiazol-2-yl)-2,5-di hydro-1H-tetrazol-5-yl] phenol**

FT-IR Spectra data for Compound (30) Show at  $3096\text{ cm}^{-1}$  for  $(\text{C}=\text{H})_{\text{arom}}$  ,  $3386\text{ cm}^{-1}$  for  $(\text{N}-\text{H})$  ,  $1642\text{ cm}^{-1}$  for  $(\text{C}=\text{N})$  ,  $1462\text{ cm}^{-1}$  for  $(\text{N}=\text{N})$  ,  $1050\text{ cm}^{-1}$  for  $(\text{C}-\text{N})$  ,  $3294\text{ cm}^{-1}$  for  $(\text{OH})$ . M.P = 178 , yield = 65 % .

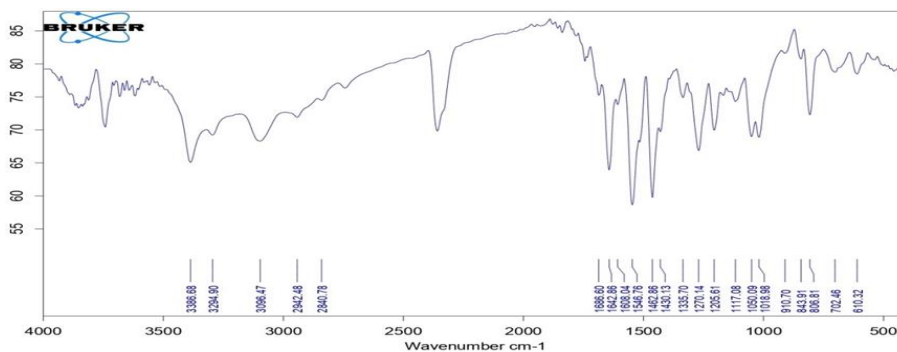
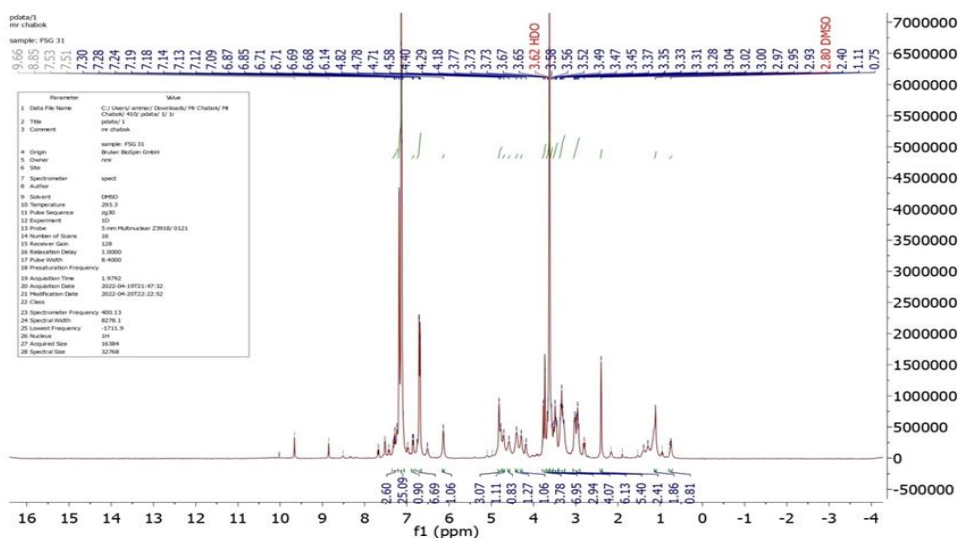
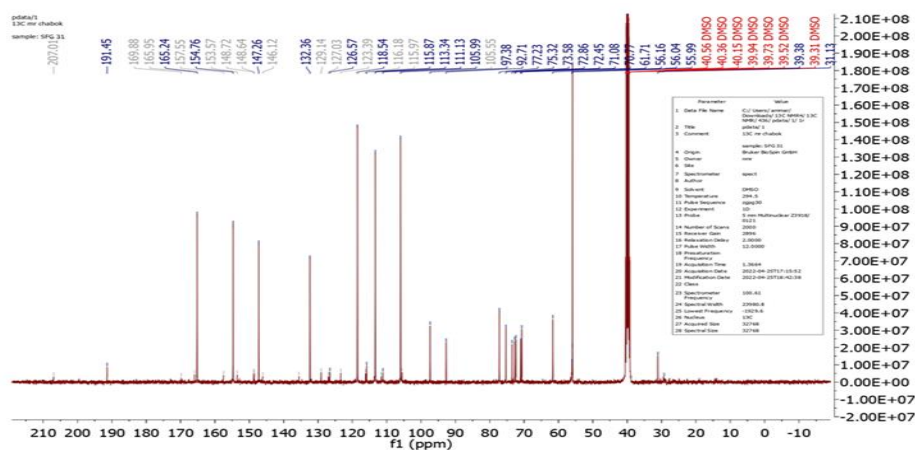


Fig (1) : FT-IR Spectra of Compound (30)

$^1\text{H}$ -NMR Spectra data for Compound (30) Show at (2.8 ppm) for DMSO , (3.6,3.4 ppm) for  $(\text{OCH}_3)$  , (6.68-7.3 ppm ) for aromatic rings ,(3.7 ppm) for  $\text{CH}-\text{N}$  , (4.7 ppm) for  $\text{N}-\text{H}$  , (9.6 ppm ) for  $\text{OH}$  .

Fig (2) :  $^1\text{H}$ -NMR Spectra of Compound (30)

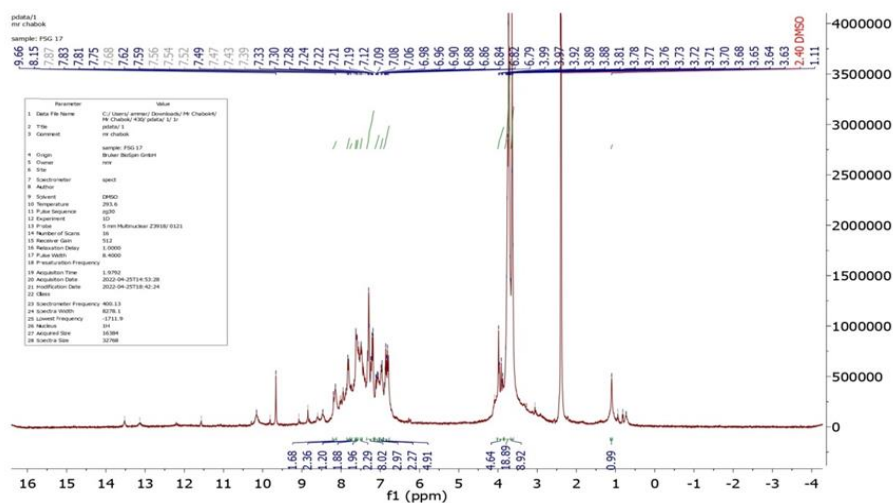
$^{13}\text{C}$ -NMR Spectra data for Compound (30) Show at (40 ppm) for DMSO , (154 ppm) for N-C=N- , (61 ppm) for C-N , (111-147 ppm) for aromatic rings , (56 ppm) O-CH<sub>3</sub> , (75 ppm) C-OH .

Fig (3) :  $^{13}\text{C}$ -NMR Spectra of Compound (30)

### Compound (31) : 2-methoxy-5-[[[6-methoxy benzothiazol-2-yl]imino][thiazol-2-yl]diazenyl]methyl]phenol

FT-IR Spectra data for Compound (31) Show at 3003  $\text{cm}^{-1}$  for (C=H)<sub>arom</sub> , 2942  $\text{cm}^{-1}$  for (C-H) , 1642  $\text{cm}^{-1}$  for (C=N) , 1597  $\text{cm}^{-1}$  for (C=C) , 1466  $\text{cm}^{-1}$  for (N=N) , 1264  $\text{cm}^{-1}$  for (C-N) , 1220  $\text{cm}^{-1}$  for (C-O) , 3081  $\text{cm}^{-1}$  for (OH) . M.P = 93 , yield = 81 % .

H<sup>1</sup>-NMR Spectra data for Compound (31) Show at (2.4 ppm) for DMSO , (3.6,3.8 ppm) for (OCH<sub>3</sub>) , (6.7-7.8 ppm ) for aromatic rings ,(9.6 ppm) for (OH) .

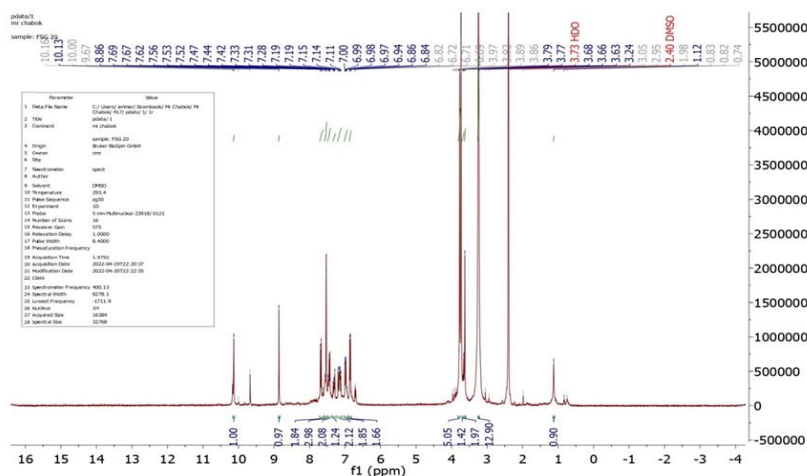


C<sup>13</sup>-NMR Spectra data for Compound (31) Show at (40 ppm) for DMSO , (56 ppm) for O-CH<sub>3</sub> , (106 ppm) for N-C=N- , (111-129.74 ppm) for aromatic rings , (155 ppm) C-OH , (114 ppm) N=C-, (166,191 ppm) for C-OCH<sub>3</sub> .

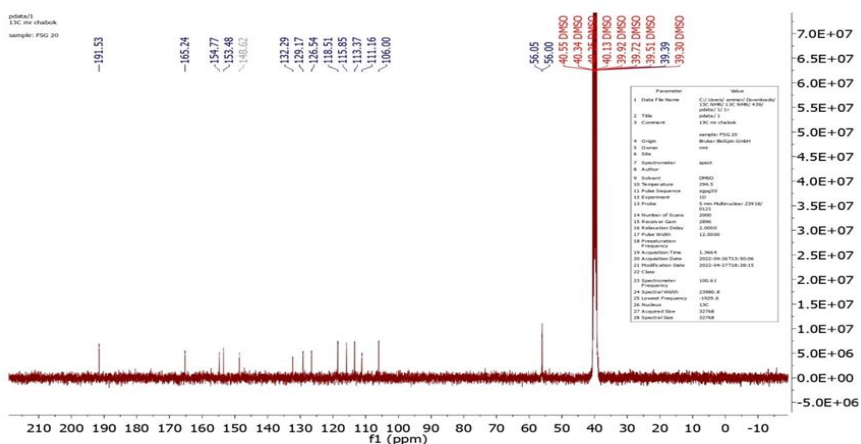
**Compound (32): 2-methoxy-5-[[[6-methoxy benzothiazol-2-yl]imino](pyridine-3-yl)diazenyl)methyl]phenol**

FTIR spectrum of the polymer film. The x-axis is Wavenumber (cm⁻¹) from 4000 to 500. The y-axis is Transmittance from 60 to 90. The spectrum shows characteristic absorption bands for the polymer. Key peaks are labeled with their wavenumbers: 3065.75, 2927.47, 2846.76, 1666.93, 1596.10, 1573.75, 1523.45, 1462.65, 1300.76, 1266.94, 1227.65, 1169.56, 1135.86, 1026.73, 822.73, 674.68, 581.60, 516.07, and 470.22.

<sup>1</sup>H-NMR Spectra data for Compound (32) Show at (2.4 ppm) for DMSO , (3.68,3.79 ppm) for (OCH<sub>3</sub>) , (6.8-8.89 ppm ) for aromatic rings ,(10.1 ppm ) for (OH) .

Fig (8) :  $^1\text{H}$ -NMR Spectra of Compound (32)

$^{13}\text{C}$ -NMR Spectra data for Compound (32) Show at (40 ppm) for DMSO , (56 ppm) for  $\text{OCH}_3$  , (106 ppm) for  $\text{N}=\text{C}=\text{N}$ - , (111-153 ppm) for aromatic rings, (113 ppm)  $\text{N}=\text{C}$ - , (165,191 ppm ) for  $\text{C}-\text{OCH}_3$  ,(154 ppm) for  $\text{C}-\text{OH}$  .

Fig (9) :  $^{13}\text{C}$ -NMR Spectra of Compound (32)

### Biological activity

The effect of the prepped derivatives was Studied on two types of Bectria (Staphylococcus aureus and Escherichia Coli ) , and the following table Shows results of this Study .

| Compounds | S.aureus | E.Coli |
|-----------|----------|--------|
| 30        | 3.2      | 3      |
| 31        | 4        | 3      |
| 32        | 3.4      | 2.6    |



Figure (10) Shows the effect of the derivatives prepared against *Staphylococcus aureus* and *Escherichia Coli* bacteria.

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