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# Synthesis and Characterization ( Oxazepine , Thiazine and Quinazoline ) Derivatives and Study the Biological Activity as Antibacterial

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**ABSTRACT**

This research Included the preparation and characterization characterization some derivatives Six and seven membered Heterocyclic Compounds ( oxazepine , thiazine , quinazoline ) The first step in clode react 2-amino-6-methoxybenzothiazole with 4- amino acetophenone to get Schiff base derivative (1). The second step react (1) with 4 -hydroxy acetophenone to get schiff base derivative (2) the last step involve react (2) with ( Phthalic , Maleic , Succinic) anhydride to get oxazepine derivatives (3,4and 5) also react (2) with(2-amino benzoic acid) and (2-mercaptobenzoic acid ) to get quinazoline (6)and thiazine (7) derivatives respectively .the physical properties of the prepared These Compounds Were Identification (FT-IR) ,(1H-NMR) and (13C-NMR) by spectroscopy then study their biological effect on two types of bacteria *Staphylococcus aureuses* (Gram positive) and *Escherichia coli* (Gram Negative)

## 1. Introduction

Heterocyclic compounds and their derivatives are considered the most biologically effective because of their great importance in the fields of medicine, as they have been used as antispasmodics, cancerous tumors, bacteria, viruses, and fungi[3]. It is part of the life structure of the components of nucleic acids, DNA, RNA, and is also involved in the synthesis of many vitamins and the manufacture of cosmetics and dyes, as well as medicines, dyes and plastics[10]. Heterocyclic compounds are also found in hemoglobin (Haem)), which transports oxygen in the blood, as well as the necessary and basic chlorophyll in the photosynthesis[13]. Schiff's bases are defined as the products of the chemical reaction of a primary amine with an aldehyde or a ketone under a given set of structural conditions. or azimethine (((C= N) with liberated water molecule which is a feature of schiff bases[7]. Is the chief bases is very important as it plays an important role in various fields such as chemistry of inorganic, organic, biological, agricultural and analytical terms are used with some metal ions as pesticides, fungi, manufacture of dyes, corrosion inhibitors and is also characterized by the biological effectiveness of high as anti-cancer[8,9]. Many researchers were able to prepare schiff bases,

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including researcher Alam S.A.M. F and his group prepared a variety of (Benz imidazole) derivatives that contain lipid bases and studied their biological effects by Studying their effectiveness on types of bacteria[1]. Also, researcher Mieaad and her group (17) were able to prepare a group of Schiff-Lekand bases and study their biological effectiveness and effect as antibacterials[10]. Also, the researcher Slassi and her group were able to prepare several types of azo-shef and study their biological activity as effective anti-fungals[13]. Oxazepine derivatives are used as anticancer and ulcerative agents[17,12]

## 2. Materials

" (FTIR) Spectra (400 -4000 $cm^{-1}$) in KBr disk were recorded on SHIMADZU FTIR-8400S Fourier transform. $^{13}$C-NMR and $^{1}$HNMR were recorded on Varian Agilent USA at (500MHz) with (DMSO-d6) measurements were made at Department of Chemistry, Tehran University, Iran."

### 2.1 Preparation of the compound (1)[15,5]

Compound No (1) prepares by react (1mg, 0.00739m mol) (4-aminoacetophenone) with (1.3333 mg, 0.00739m mol ) of (2-amino -6- methoxybenzothiazole ) in (25 ml ) Ethanol. with, (3 drops) glacial acetic acid(99%), after which the reflux is done at a temperature of ($78^0$ C) for a period of (22 hours), and then the solution is left to cool at room temperature for leave. For a period of (24) hours , after then recrystallized .The course of the reaction was monitored By use, technique thin layer chromatography (TLC)

### 2.2 Preparation of the compound (2)

The compound (2) Schiff base was prepared by reacting (1 mg , 0.00336m mol ) of compound (1) with (0.456 mg, 0.00336m mol) of (4- hydroxy-acetophenone) in (25 ml) of ethanol and then (3 drops) ) of glacial acetic acid, and reflux at a temperature of ($78^0$ C) for a leave of (26 hours), after which the solution is left to cool at room temperature for a period of (24) hours, and the sediments are recrystallized With methanol

### 2.3 Preparation of compound(Oxazepine) (general metode ) (3,4,5)[4]

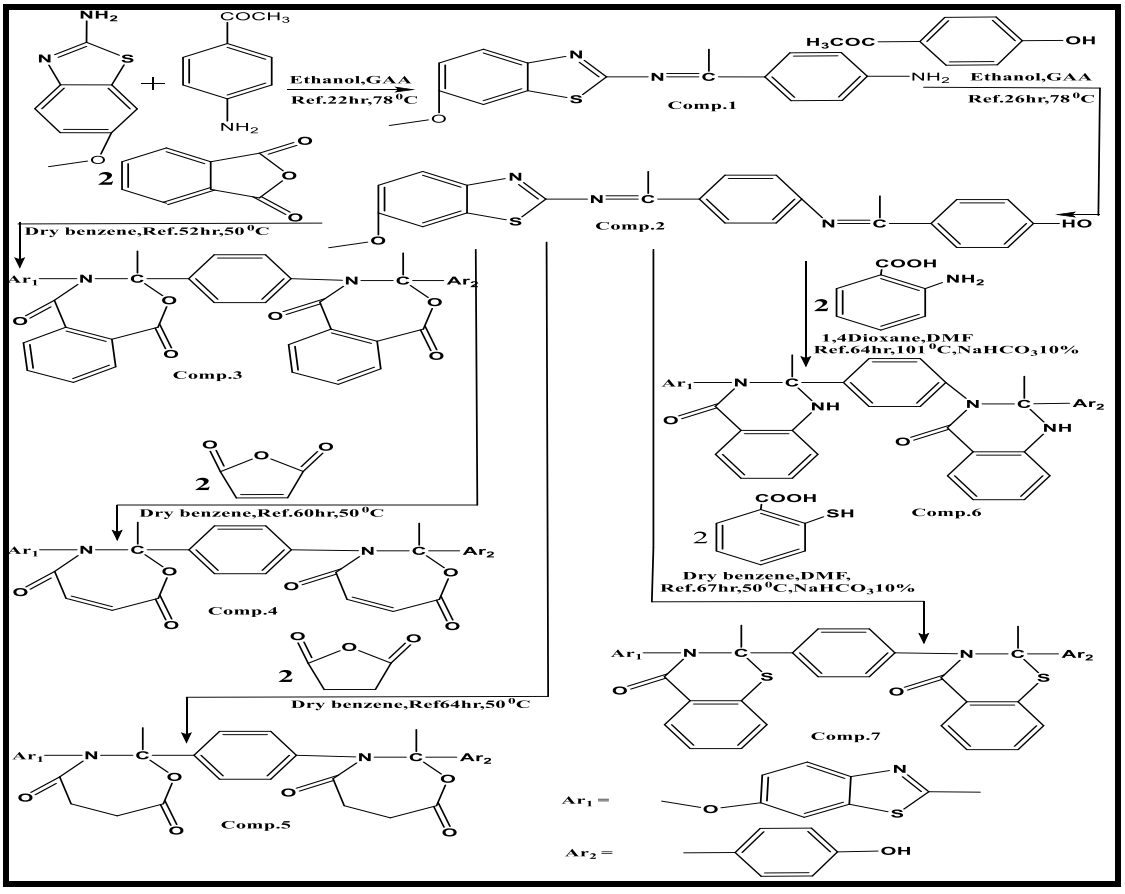
The three derivatives (Oxazepine) were prepared by reacting (0.6 mg, 0.001444mmol) of compound (2) with each of (0.4277 mg, 0.001444m mol), (phathalic anhydride), (0.2831mg, 0.001444m mol) (maleic anhydride), ( 0.289mg,0.001444mmol) (Succinic anhydride) each dissolves in (30 ml) of dry benzene. The reflux was done from (52-64) hours at a temperature of ($80^0$ C) after that the solution is left to cool down for a period of (24 hours), After that, it is filtered and recrystallized with ethanol

### 2.4 Preparation of compound (6) [16]

Compound (6) is prepared by adding (0.6 mg ) (0.001444m mol ) of compound (2) dissolved in (20) ml (1.4 dioxane) to (0.369mg) (0.001444m mol) of (2-amino benzoic acid) by adding (3 ml) ) Of DMF and then it is reflux (64) hours at a temperature of ($101^0$ C), after and 10% of sodium bicarbonate solution is added, then it is filtered and then recrystallized with a mixture of (ethanol - water) By (1:2)

### 2.5 Preparation of compound (7) [17]

Compound (7) is prepared by adding (0.6m g) (0.001444mmol ) of compound (2) and dissolved in (22 ml) (benzene) to 0.4452 mg,0.001444m mol) from 2-mercapto benzoic acid ) with the addition of ( 3 ml) of (DMF) and (5) drops of (Tri ethyl amine), after which it is escalated for a period of (55) hours at a temperature of (50$^0$C) added (10%) of sodium bicarbonate solution, then filtering and then recrystallizing with (1,4-Dioxan)



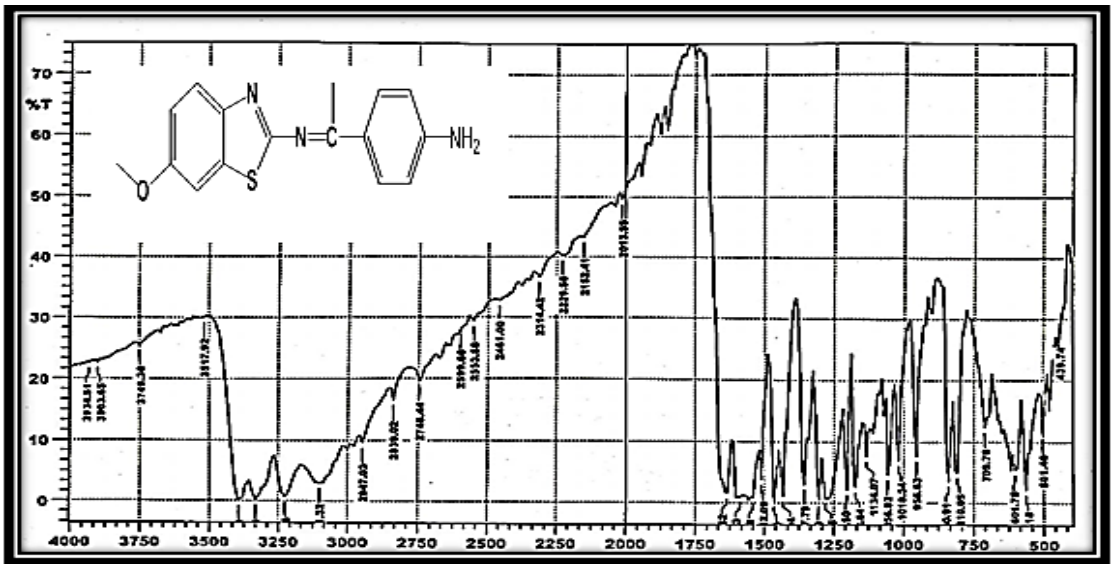

**scheme(1) prepare of some heterocyclic derivatives**

## 3. Results and Discussion

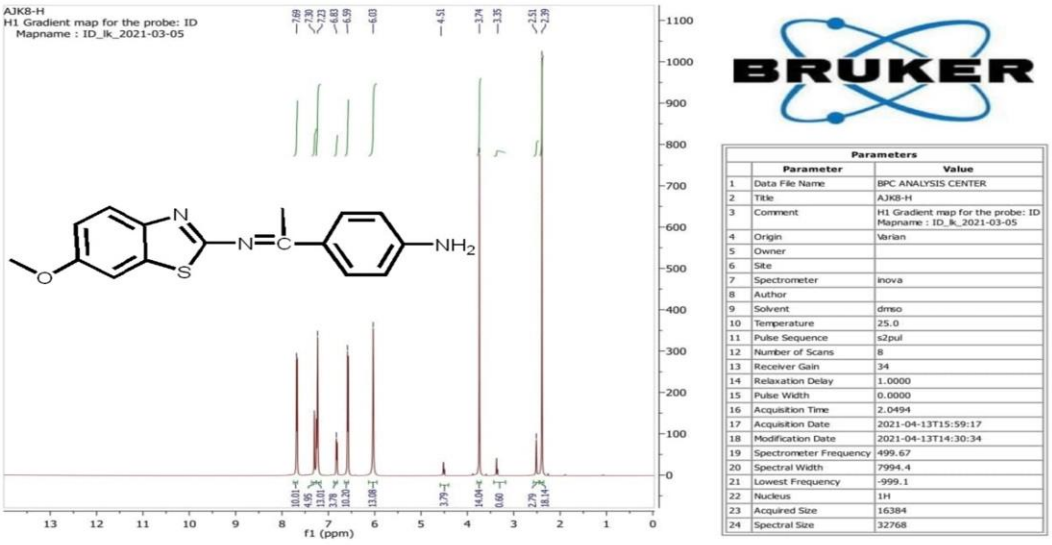
### Characterization of the compound (1)

### 4-(1-((6-methoxybenzo[d]thiazol-2-yl)imino)ethyl)aniline

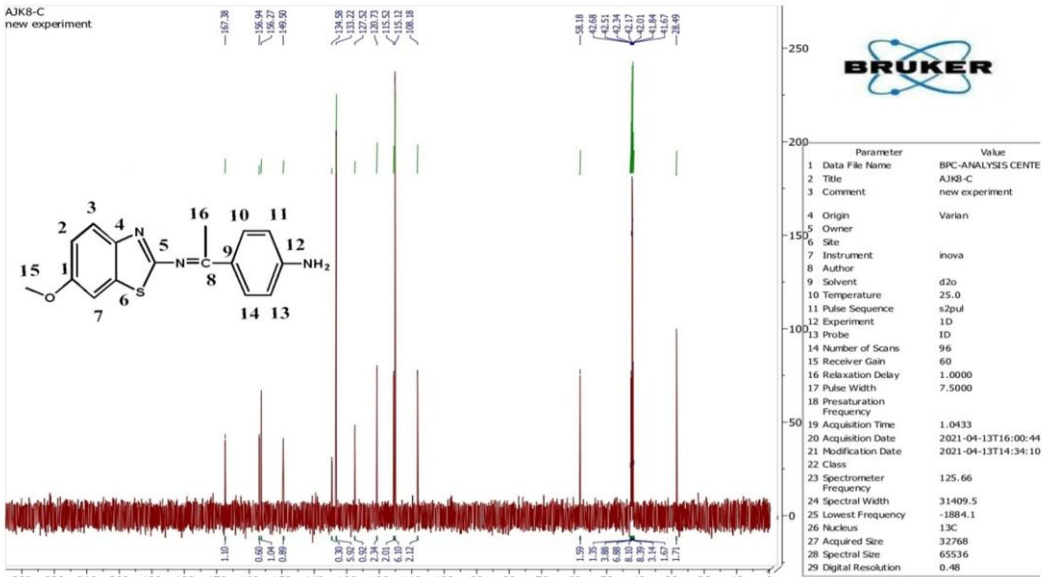
FT-IR spectrum data for the compound (1) show two peaks at (3491-3353)cm-$^1$for (-NH2) , peak at 3101 cm$^{-1}$ fo r (Ar – H) , 2947 cm$^{-1}$ for (C- H) in CH3 , 1649 cm$^{-1}$ for (C=N), 1621 cm$^{-1}$ fo r (C=C),1272cm$^{-1}$for (C-S). $^1$HMNR spectrum data of compound (1) show 2.50ppm (DMSO) , 4.5ppm (S ,2H, (NH2)) , 3. 7ppm (S ,3H, (OCH3)) , 3.3 ppm (S ,3H, (CH3)( 6.03-7.69 ppm (m ,7H, (Ar-H)), . The$^{13}$ CNMR spectrum data ( DMSO) compound (1) show : 58 ppm ($C_{15}$ ) ,167 ppm($C_5$), 156ppm ($C_8$), 28ppm ($C_{16}$) 108-149ppm ($C_{Arom}$)




**Fig(1) FTIR spectrum of compound(1)**




**Fig(2)($^1$ H-NMR) spectrum of compound(1)**

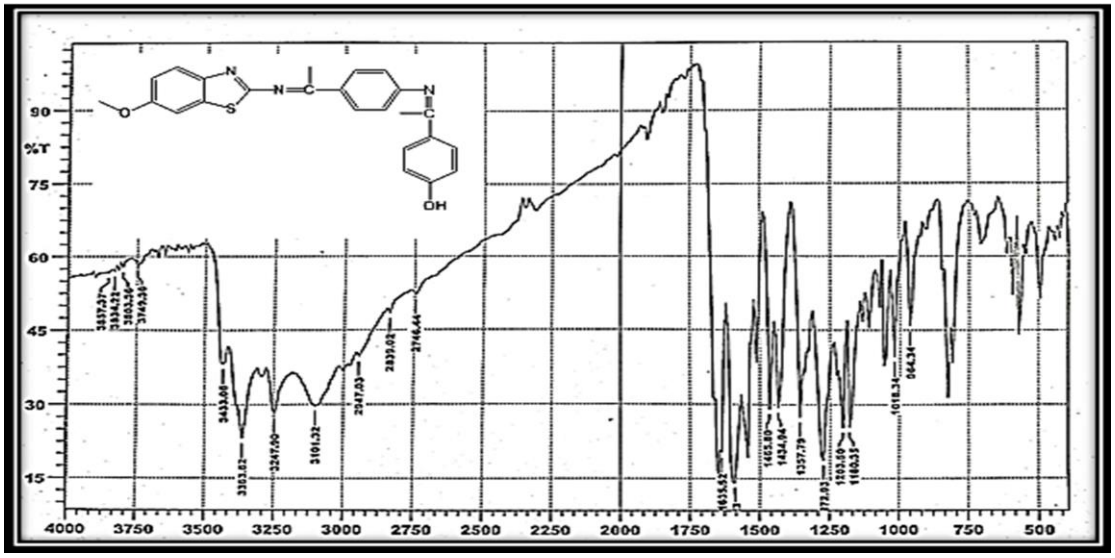



**Fig(3) ($^{13}$C-NMR) spectrum of compound(1)**

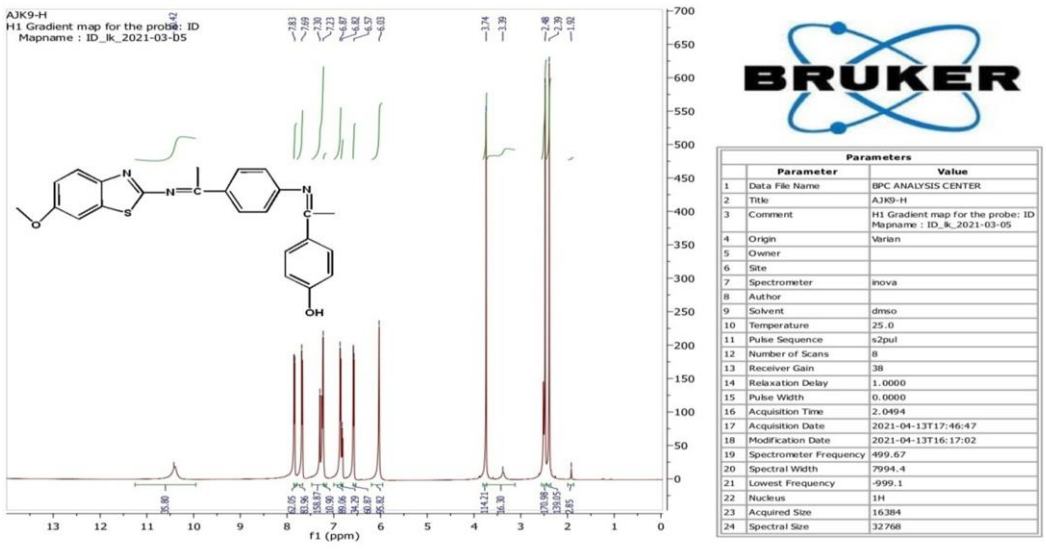
## Characterization of the compound (2)

### 4-(1-((4-(1-((6-methoxybenzo[d]thiazol-2-yl)imino)ethyl)phenyl)imino)ethyl)phenol

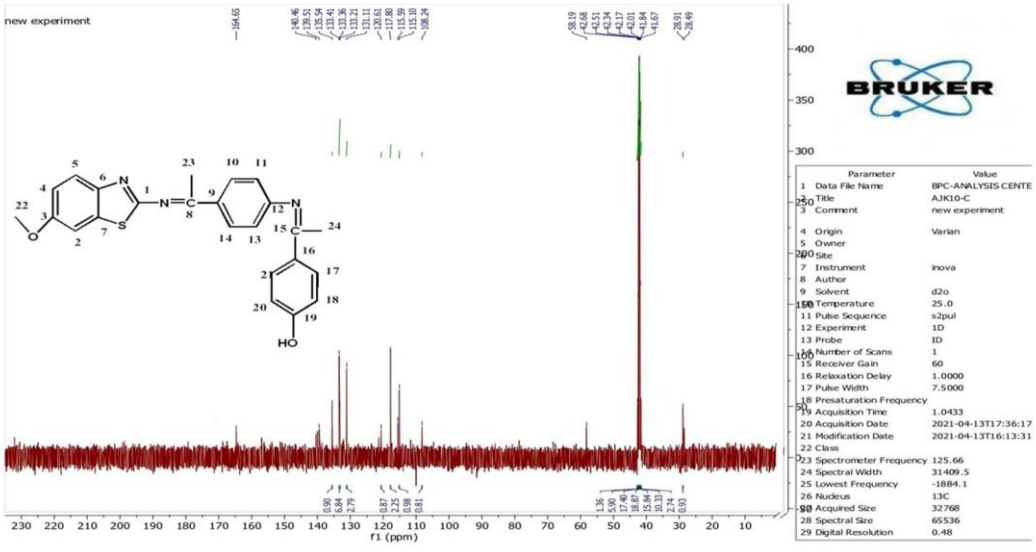
FT-IR spectrum data for the compound (2) at 3433 $cm^{-1}$ for (O H) , 3101 $cm^{-1}$ for (Ar – H) , 2947$cm^{-1}$ for (C- H) in CH3 , 1635 $cm^{-1}$ for (C=N), 1602 $cm^{-1}$ for (C=C),1272 cm-1for (C-S). $^{1}$HMNR spectrum data of compound (2) show 2.50ppm (DMSO) , 10.4ppm (S ,1H, (OH)) , 3.7ppm (S ,3H, (OCH3)) , 1. 9ppm (S ,6H, (CH3)), 6.0-7.8 ppm (M ,18H, (Ar-H)),. The$^{13}$ C-NMR spectrum data ( DMSO) compound (2) show : 58 ppm ($C_{22}$ ) ,28 ppm($C_{23}$,$C_{24}$), 164ppm ($C_1$) , 139ppm ($C_{15}$, $C_8$) , , 108 -140ppm ($C_{Arom}$)




**Fig(4) FTIR spectrum of compound(2)**




**Fig(5)($^{1}$ H-NMR) spectrum of compound(2)**



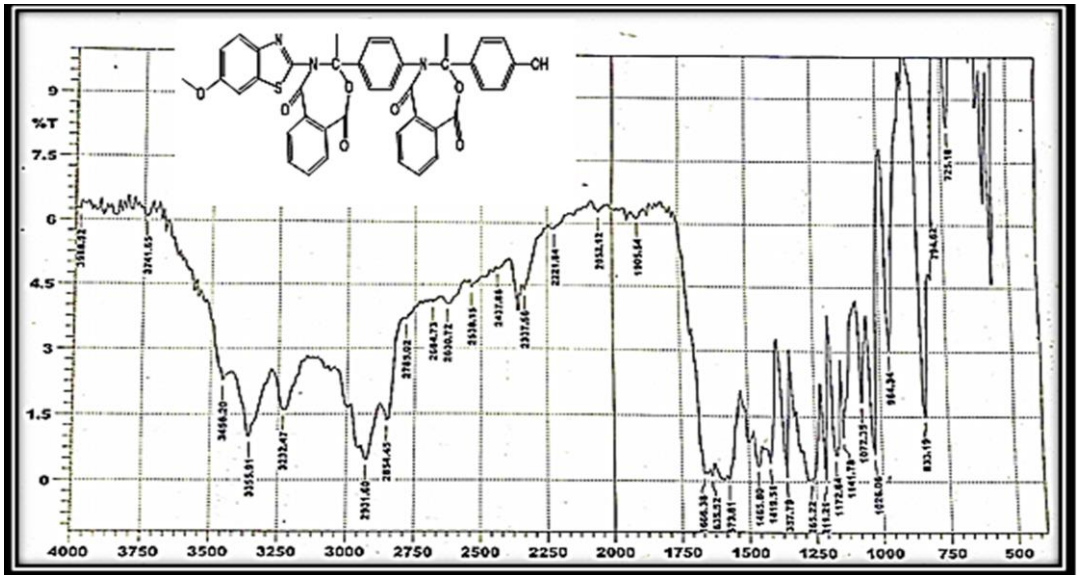

**Fig(6) ($^{13}$C-NMR) spectrum of compound(2)**

## Characterization of compound (3)

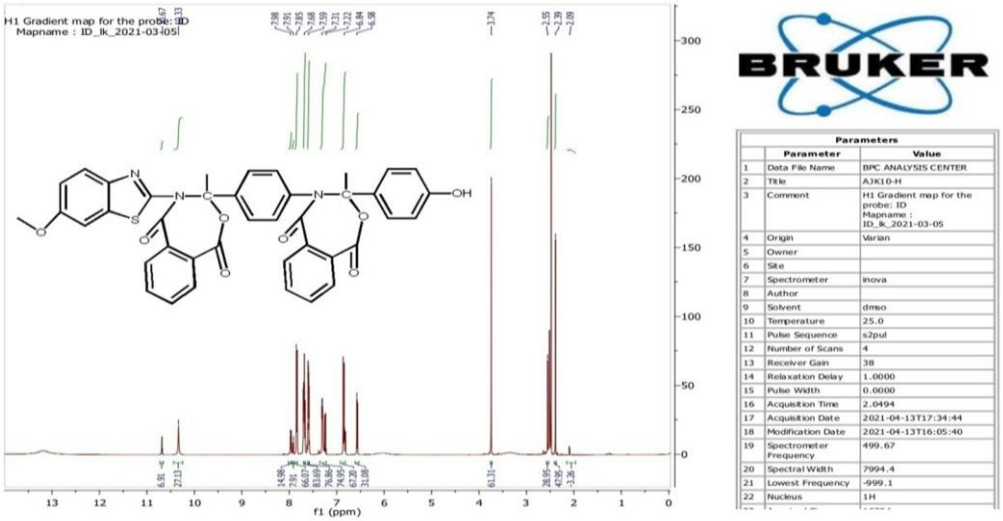
**3-(4-hydroxyphenyl)-4-(4-(4-(6-methoxybenzo[d]thiazol-2-yl)-3-methyl-1,5-dioxo-1,3,4,5-tetrahydrobenzo[e][1,3]oxazepin-3-yl)phenyl)-3-methyl-3,4-dihydrobenzo[e][1,3]oxazepine-1,5-dione**

FT-IR spectrum data for the compound (3) show peak at 3355 $cm^{-1}$ for (OH) 3232 $cm^{-1}$ for (Ar – H) , 2931 $cm^{-1}$ for (C- H) in CH3 ,1666$cm^{-1}$ for (C=O), 1635 $cm^{-1}$ for (C=N), 1573 $cm^{-1}$ for (C=C), 1265 $cm^{-1}$for (C-S). $^{1}$HMNR spectrum data of compound (3) show 2.50ppm

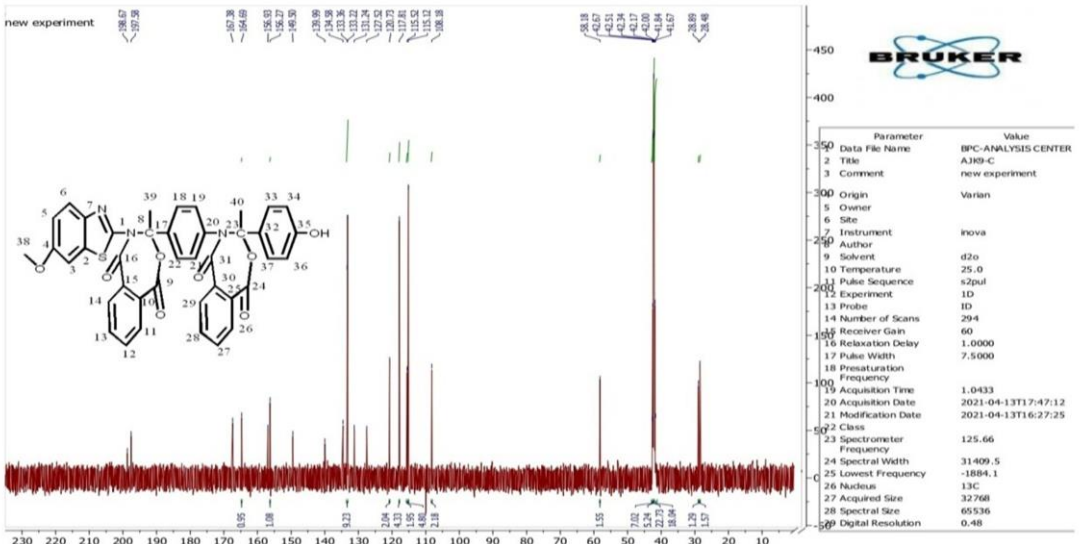
(DMSO) ,10.3(S,1H,(OH)), 3.7ppm (S ,3H, (OCH3)) , 2.09ppm (S ,6H, (CH3)), 6.5-7.9ppm (M ,11H, (Ar-H)),. The$^{13}$ CNMR spectrum data ( DMSO) compound (3) show : 197 ppm ($C_{16}$,$C_{31}$ ) ,198 ppm($C_9$,$C_{24}$), 28.4ppm ($C_{40}$,$C_{39}$) , 167ppm (C1) , 58ppm ($C_8$,$C_{23}$) , 164ppm($C_{35}$) .,28.8ppm( $C_{38}$),( 108-156)($C_{Arom}$)




**Fig(7) FTIR spectrum of compound(3)**




**Fig(8)($^1$ H-NMR) spectrum of compound(3)**

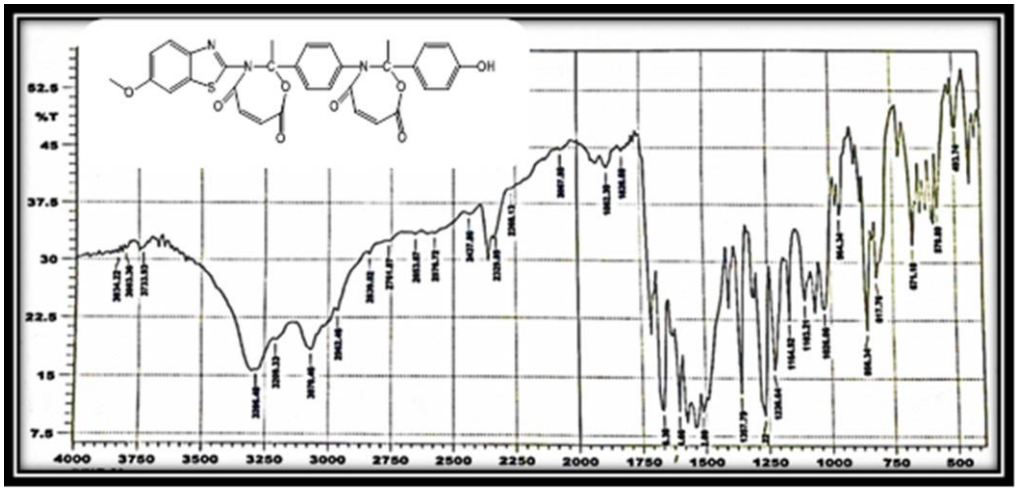



**Fig(9) ($^{13}$C-NMR) spectrum of compound(3)**

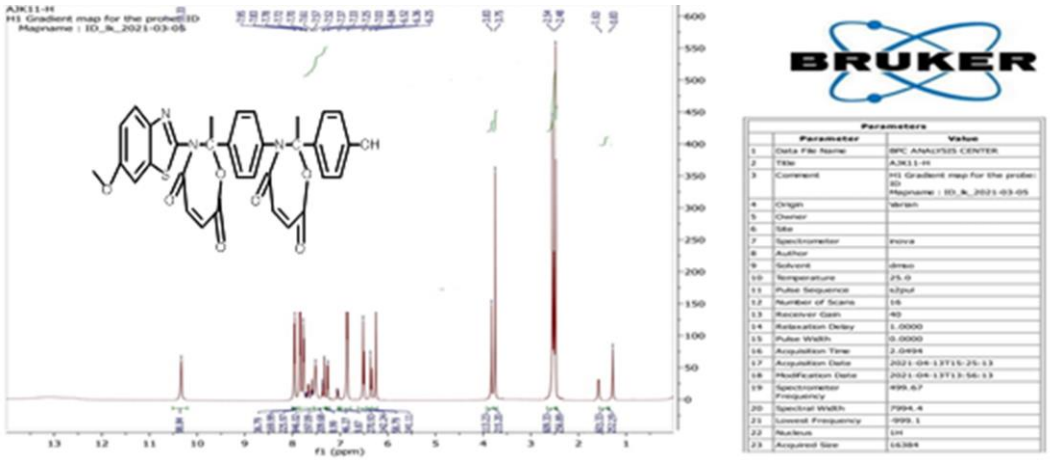
**Characterization of compound (4)**

**2-(4-hydroxyphenyl)-3-(4-(3-(6-methoxybenzo[d]thiazol-2-yl)-2-methyl-4,7-dioxo-2,3,4,7-tetrahydro-1,3-oxazepin-2-yl)phenyl)-2-methyl-2,3-dihydro-1,3-oxazepine-4,7-dione**

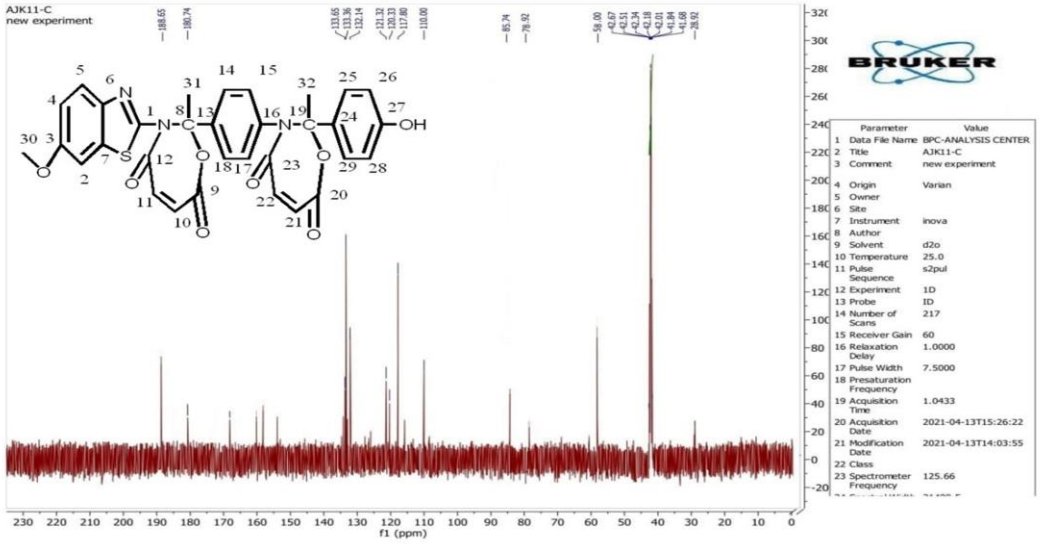
FT-IR spectrum data for the compound (4) show peak at3286cm$^{-1}$ for (OH), 3070cm$^{-1}$ for (Ar – H) , 2962cm$^{-1}$ for (C- H) in CH3 , 1636cm$^{-1}$for (C=O), 1604 cm$^{-1}$ for (C=N), 1506 cm$^{-1}$ for (C=C),1226 cm$^{-1}$ for (C-S). $^{1}$HMNR spectrum data of compound (4) show 2.50ppm (DMSO) 10.3(1H)(OH),3.7(2H) (C=O)-CH) ,3.8(2H)(O-(C=O)CH), , 2.4ppm (S ,3H, (OCH3)) , 1.2,0.8ppm (S ,6H, (CH3)), 6.2-7.9 ppm(11H)(Ar-H) . The $^{13}$ C-NMR spectrum data ( DMSO) compound (4) show : 28 ppm ($C_{31}$,$C_{32}$ ) ,85 ppm($C_{11}$,$C_{22}$), 80ppm ($C_{10}$,$C_{21}$) , 58ppm ($C_{30}$) , 168ppm ($C_1$), 191ppm($C_{12}$,$C_{23}$) ,180ppm( $C_9$,$C_{20}$),154 ppm(C30), 110-150($C_{Arom}$)




**Fig(10) FTIR spectrum of compound(4)**



**Fig(11)($^{1}$ H-NMR) spectrum of compound(4)**



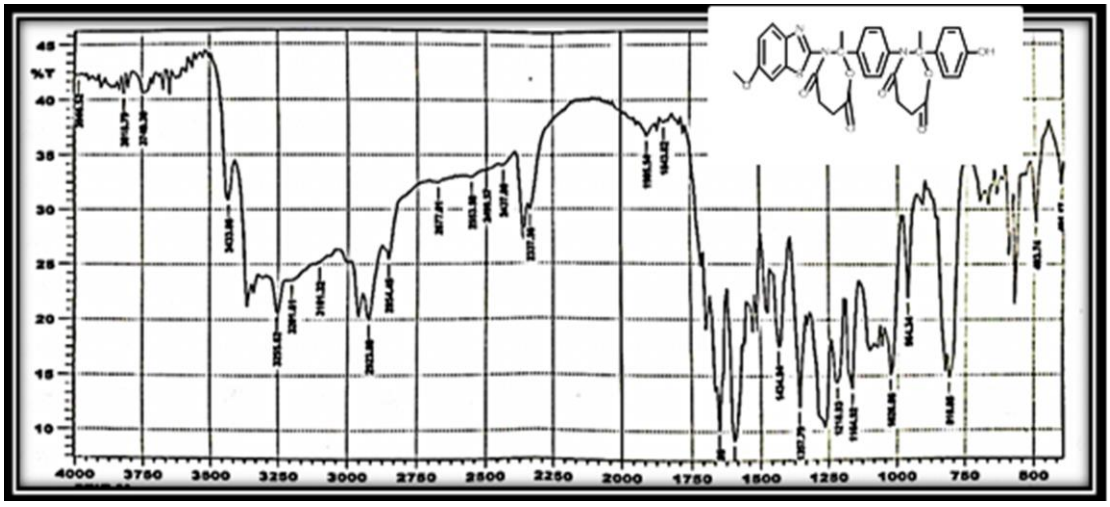
**Fig(12) ($^{13}$C-NMR) spectrum of compound(4)**

**Characterization of compound (5)**

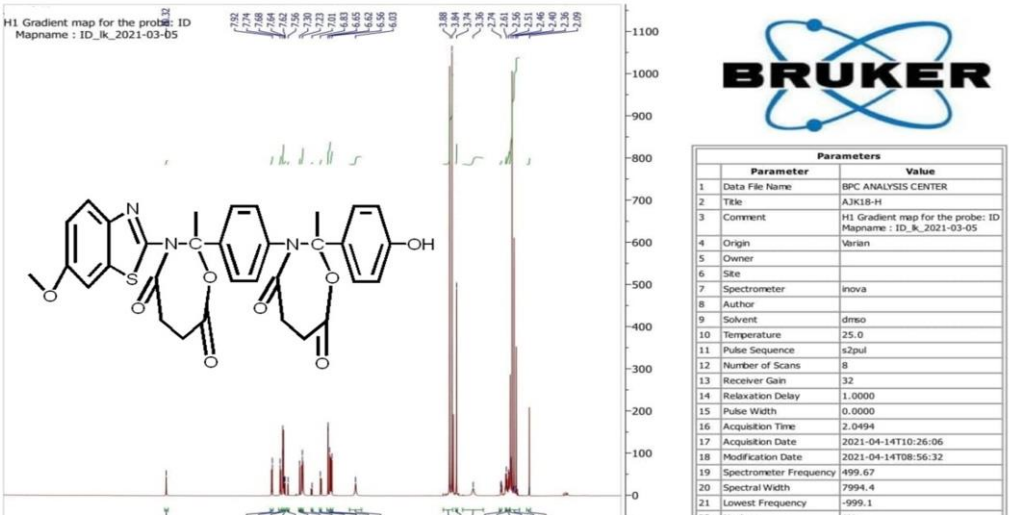
**2-(4-hydroxyphenyl)-3-(4-(3-(6-methoxybenzo[d]thiazol-2-yl)-2-methyl-4,7-dioxo-1,3-oxazepan-2-yl)phenyl)-2-methyl-1,3-oxazepane-4,7-dione**

FT-IR spectrum data for the compound (5) show peak at3322cm$^{-1}$ for (OH), 3255cm$^{-1}$ for (Ar – H) , 2923cm$^{-1}$ for (C- H) in CH3 ,1656cm$^{-1}$for (C=O), 1608 cm$^{-1}$ for (C=N), 1434 cm$^{-1}$ for (C=C),1226 cm$^{-1}$for (C-S). $^{1}$HMNR spectrum data of compound (5) show 2.50ppm (DMSO) , 2.6ppm (S ,3H, (OCH3)) , 2.09ppm (S ,6H, (CH3)),10.3(1H)(OH), 3.7,3-8 ppm (4H)(O-(C=H)-CH) ,3.7.2.7(4H)(C=O)C-H ,6.0-7.9 ppm (11H,Ar-H)). The$^{13}$ CNMR spectrum data ( DMSO) compound (5) show : 11 ppm ($C_{31}$,$C_{32}$ ) ,28.4 ppm($C_{11}$,$C_{22}$), 28.8ppm

($C_{10}$,$C_{21}$) , 38ppm ($C_{30}$) , 48ppm ($C_{19}$) , 58ppm ($C_8$) 197ppm($C_9$,$C_{20}$) , 198ppm($C_{23}$,$C_{12}$),164,108 ppm($C_{Arom}$) .




**Fig(13) FTIR spectrum of compound(5)**

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| 6 | Site | |
| 7 | Spectrometer | inova |
| 8 | Author | |
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| 10 | Temperature | 25.0 |
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| 18 | Modification Date | 2021-04-14T08:56:32 |
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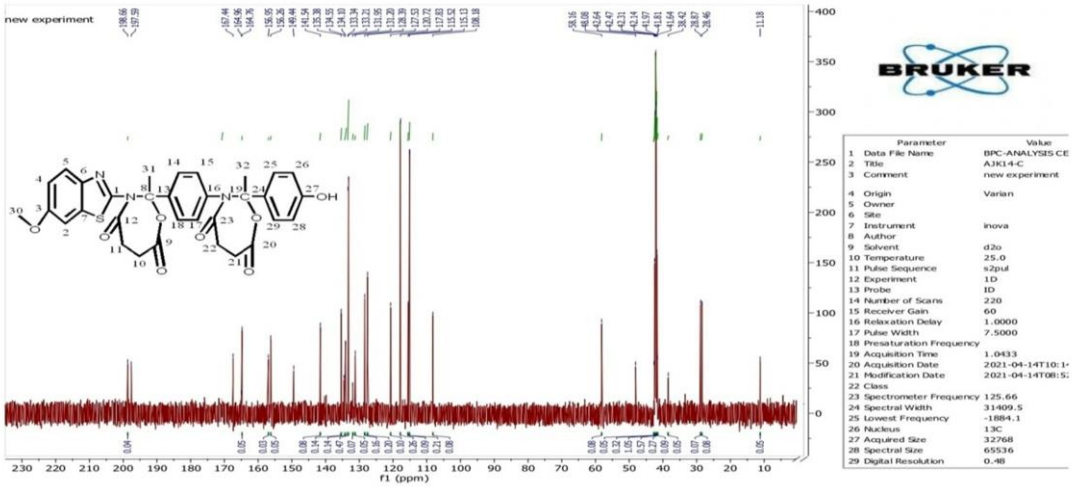
**Fig(14)($^1$ H-NMR) spectrum of compound(5)**

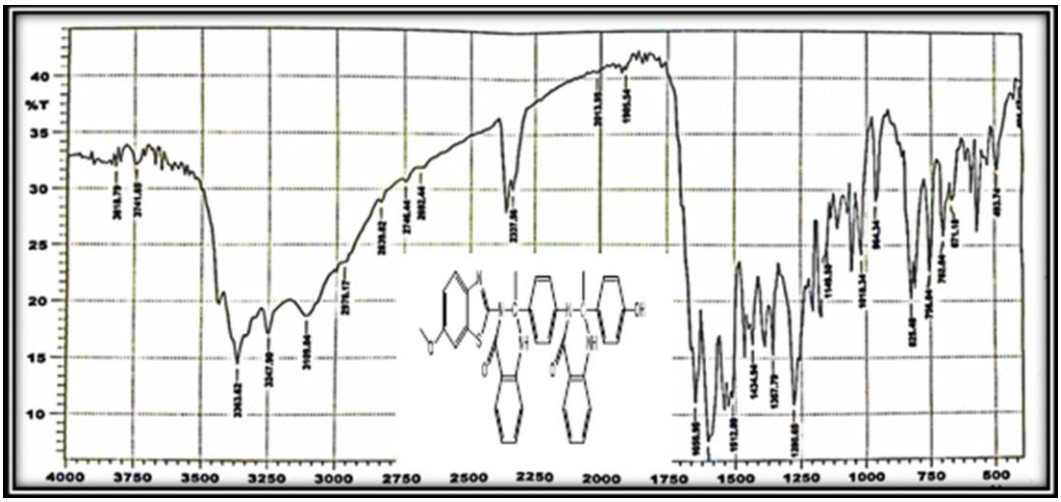



**Fig(15) ($^{13}$C-NMR) spectrum of compound(5)**

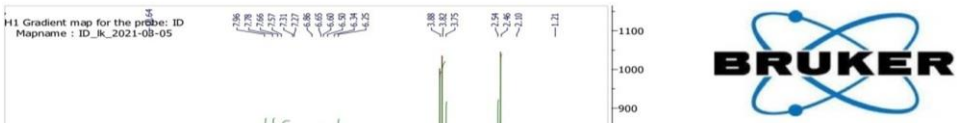
**Characterization of compound (6)**

**2-(4-hydroxyphenyl)-3-(4-(3-(6-methoxybenzo[d]thiazol-2-yl)-2-methyl-4-oxo-1,2,3,4-tetrahydroquinazolin-2-yl)phenyl)-2-methyl-2,3-dihydroquinazolin-4(1H)-one**

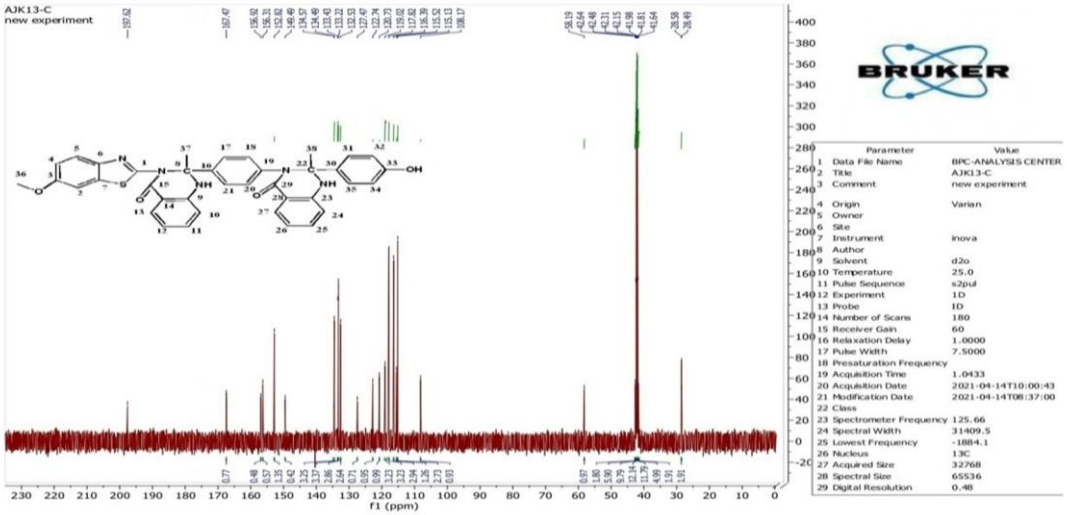
FT-IR spectrum data for the compound (6) show peak at3247cm$^{-1}$ for (OH) 3109cm$^{-1}$ for (Ar – H) , 3363cm$^{-1}$ for (-NH),2970cm$^{-1}$ for (C- H) in CH3 ,1650cm$^{-1}$ for (C=O), 1605 cm$^{-1}$ for (C=N), 1512cm$^{-1}$ for (C=C),1280 cm$^{-1}$ for (C-S). $^{1}$HMNR spectrum data of compound (5) show 2.50ppm (DMSO) , 3.8 ppm (S ,2H,NH) , 3.7ppm (S ,3H, (OCH3)), 1.2,2. 1 ppm (S,6H, CH3), 6.2-7.9ppm (M , 19H,Ar-H)),10.6(1h)(OH). The $^{13}$ CNMR spectrum data ( DMSO) compound (6) show : 197 ppm ($C_{15}$,$C_{29}$) ,28.4ppm($C_{37}$,$C_{38}$), , 28.2ppm ($C_{36}$) , 58ppm ($C_{9}$,$C_{22}$) , .167ppm($C_{1}$), ( 108-156)($C_{Arom}$).




**Fig(16) FTIR spectrum of compound(6)**



**Fig(17)($^{1}$ H-NMR) spectrum of compound(6)**



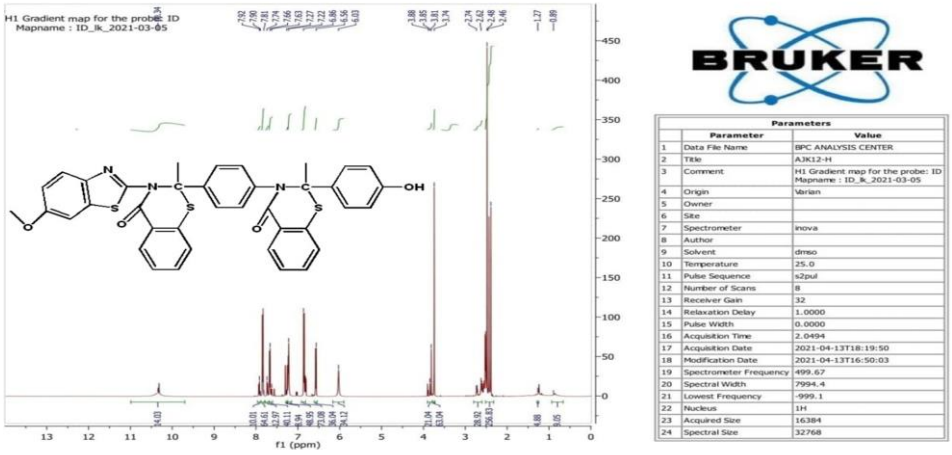

**Fig(18) ($^{13}$C-NMR) spectrum of compound(6)**

**Characterization of compound (7)**

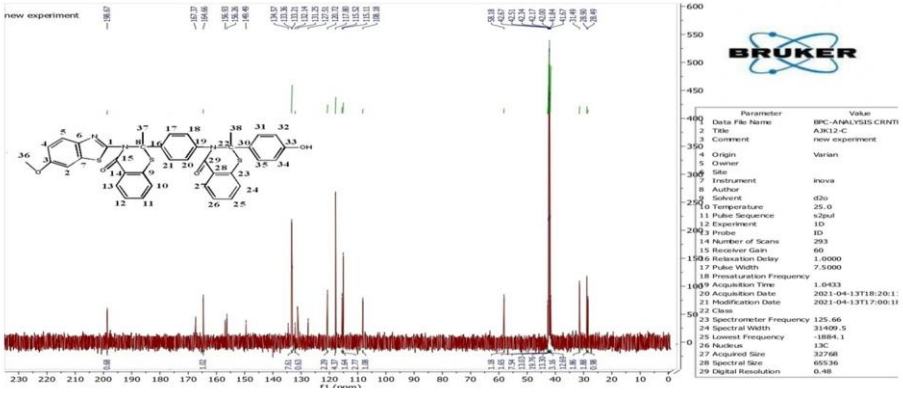
**2-(4-hydroxyphenyl)-3-(4-(3-(6-methoxybenzo[d]thiazol-2-yl)-2-methyl-4-oxo-3,4-dihydro-2H-benzo[e][1,3]thiazin-2-yl)phenyl)-2-methyl-2,3-dihydro-4H-benzo[e][1,3]thiazin-4-one**

FT-IR spectrum data for the compound (7) show peak at 3440cm$^{-1}$ for (OH) 3078cm$^{-1}$ for (Ar – H) , 2923cm$^{-1}$ for (C- H) in CH3 , 1650cm$^{-1}$for (C=O), 1564cm$^{-1}$ for (C=C),1253 cm$^{-1}$for (C-S). $^{1}$HMNR spectrum data of compound (7) show 2.52ppm (DMSO) , 3.8ppm (S ,3H, (OCH3)) , 0.8- 1.2ppm (S ,6H, (CH3)), 6.0-7.9 ppm (M ,19H, (Ar-H)), 10.3 ppm (1H)(OH) .The$^{13}$ CNMR spectrum data ( DMSO) compound (7) show : 198 ppm ($C_{15}$,$C_{29}$) ,28.4,28.9ppm($C_{37}$,$C_{38}$), 31ppm ($C_{36}$) , 58ppm ($C_{8}$,$C_{22}$) , 167ppm ($C_{1}$) , , 108-164 ppm 9($C_{Arom}$)

**Fig(19) FTIR spectrum of compound(7)**




**Fig(20)([1] H-NMR) spectrum of compound(7)**




**Fig(21) ([13]C-NMR) spectrum of compound(7)**

## 4. Biological activity

### 4.1 antibacterial

The results show that derivetives reduce significant antibacterial effectiveness. against bacteria "*staphylococcus aurous* And *Escherichia coli*" . the compounds. That show good activity are (1,2,3,4,5,6,7) against (*staphylococcus aurous*) , and compound that show very good activity are (2) against (*staphylococcus aurous*) , the results of the antibacterial activity are shown in the fig (22)

| Comp.NO | Staph aureus | Mm | E.Coli | Mm |
|---|---|---|---|---|
| 1 | ++ | 15 | +++ | 22 |
| 2 | +++ | 21 | ++ | 22 |
| 3 | ++ | 14 | ++ | 11 |
| 4 | ++ | 14 | ++ | 19 |
| 5 | + | 7 | ++ | 16 |
| 6 | ++ | 15 | + | 14 |
| 7 | +++ | 20 | ++ | 13 |

**Table (1) the results of the antibacterial activity for (1-7) derivatives**

"+= (5-10)mm =slightly active, ++= (11-20)mm moderately, +++ = More than 20 , good active"

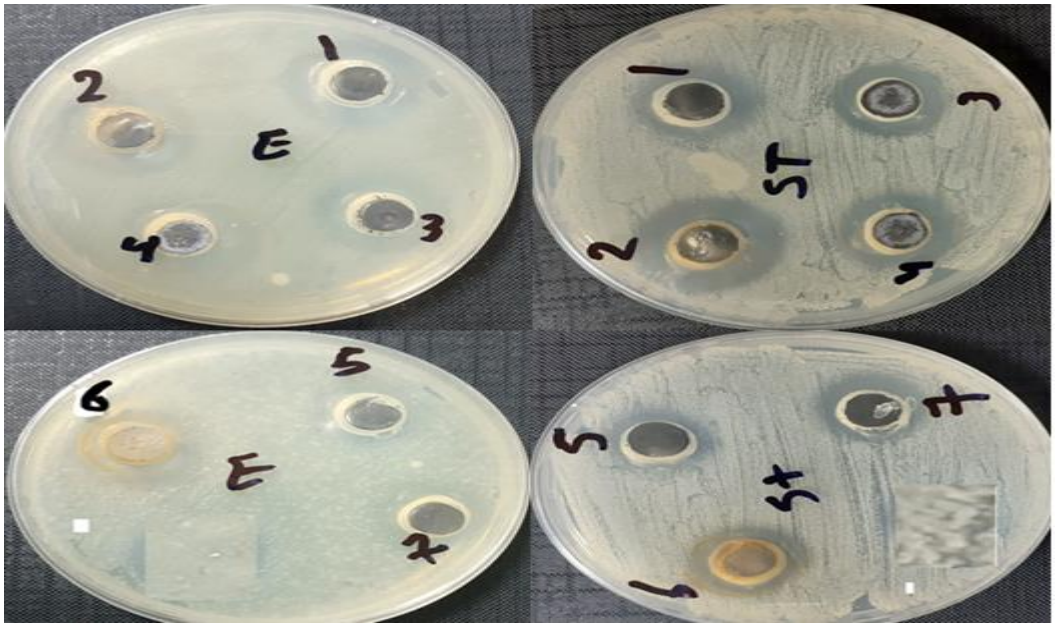


**Fig (22) effect compounds (*Staph aurous*) aginst and (*E.Coli*) aginst**

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