

# Synthesis And Characterization Of New Azetidinone Ring Derivative From Sulphadiazine Drug

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**الخلاصة:** تضمنت الدراسة تحضير مشتق حلقة الازيتيديون الجديد من مركب الازو المحضر سابقا (N1) [4-Amino-3--((4-methoxy-2-nitrophenyl)diazenyl)-N-(Pyrimidine-2-yl)benzen-esulfonamide] بواسطة تفاعله مع بارا-أمينو اسيتوفينون ليعطي قاعدة شيف (I). من مجموعة الايمين ومجموعة الامين الاروماتية الحرة في قاعدة شيف (I). حضر المشتق الحاوي على حلقات سباعية ورباعية غير متجانسة بعدة خطوات. أغلب هذه المشتقات أثبتت من خلال أطيف الأشعة تحت الحمراء وأطيف رنين البرتون والكربون المغناطيسي.

الكلمات المفتاحية: تفاعل ستدنكر, ازيتيديون, سلفاديازين, اوكسازيبين.

## Abstract

The study Included synthesis of some new azetidinone ring Derivatives from prepared Azo Compound [4-Amino-3--((4-methoxy-2-nitrophenyl)diazenyl)-N-(Pyrimidine-2-yl)benzen-esulfonamide](N1) by the reaction of it with p-amino acetophenone " to give the Schiff base (I). From the imine group and free aromatic amine group in the Schiff base (I), a derivative containing seven and four rings was prepared in several steps. Most of these derivatives were confirmed by "FT-IR, <sup>1</sup>HNMR and <sup>13</sup>CNMR" spectra.

Keywords: Staudinger reaction, Azetidinone, Sulphadiazine, Oxazepine.

## Introduction:

[2+2] cycloaddition or The Staudinger reaction is a reaction between imine and ketene which Represent as one of the most essential and flexible strategies for the synthesis of structurally varied derivatives of 2-azetidinone[1]. by using acid chlorides in the presence of (Et<sub>3</sub>N) triethylamine or α-diazoketones as precursors for ketene, The Staudinger reaction gets thermally or photochemically[2]. Azetidinone is a four-membered cyclic has been known as a beneficial building block for the preparation of numerous of organic compounds by take advantage of the strain energy that linked with it[3]. Sulphadiazine is a sulfonamide antibiotic and it is recognized as one of " the World Health Organization's List of Essential Medicines". It removes bacteria that causes infections by stopping the production of folic acid into the bacterial cell, and is usually used to treat " urinary tract infections" (UTIs) and burns[4,5]. 1,3-Oxazepine is unsaturated cyclic compound of seven atoms, containing an oxygen replacing carbon No.1 and a nitrogen replacing carbon No.3. Prepared by the pericyclic cycloaddition of schiff bases with phthalic, nitro phthalic, succinic and maleic anhydrides [6,7]. " 2-Azetidinone" also known "β-lactam" are four-membered cyclic amide derived from 3-amino-propanoic acid[8,9]. The parent heterocyclic ring of azetidinone is azetidine that is a four member heterocyclic ring system with (N) as hetero atom. 2-Azetidinone includes a carbonyl group on the second position which is one of the most common heterocyclic rings found in many antibiotics[10]. Although the ring of azetidinone was known since (1907) but the

realization of their chemistry began from (1947) only. These are presently used for chemotherapy of bacterial infections[11-13].

#### **MATERIALS AND METHODS**

The melting points were recorded and expressed in degree ( $^{\circ}\text{C}$ ) by using the electro thermal 9300 melting point LTD, UK. Thin layer chromatography T.L.C was performed on aluminum and glass plates coated with 0.25mm layer of silica-gel (Fluka). Some of the derivatives were detected by iodine vapor. FT-IR spectra, Fourier transform infrared (SHIMADZU, 8400) spectrophotometer, Japan the range 4000-600 $\text{cm}^{-1}$ . The samples were run in KBr disc.  $^{13}\text{C}$ ,  $^1\text{H}$ -NMR spectra in (ppm) unit were operating in DMSO-*d*<sub>6</sub> as solvent using (Bruker- Ultra Shield 300 MHz Switzerland).

Synthesis of Basic compound(Azo) (N1) **[4-Amino-3-((4-methoxy-2-nitrophenyl) diazenyl)-N-(Pyrimidine-2-yl)benzenesulfonamide]** according to the previously paper[3].

**Synthesis comp. (I)[4-((Z)-1-(4-aminophenyl)ethylideneamino)-3-(E)-(4-methoxy-2-nitrophenyl)diazenyl)-N-(pyrimidin-2-yl)benzenesulfonamide][14].**

Azo Compound (N1) (0.43gm, 0.001 mol) was dissolved in hot concentration glacial acetic acid about (50 ml) then added to (0.164gm, 0.001 mol) of p-amino aceto phenone was dissolved in (5 ml) of glacial acetic acid. The reaction mixture was refluxed at ( $100^{\circ}\text{C}$ ) with stirring for (50) hour. The progress of the reaction was followed by T.L.C. After the completion the mixture was poured onto ice crushed. The yielded solid was filtered off and wash with (2%) Sodium bicarbonate solution and distilled water then recrystallized from abs. Ethanol. Yield, Dark red, (67%), m.p. ( $80-84^{\circ}\text{C}$ ) and ( $R_f = 0.52$ ) (Met: Tol)(2:3).

**FT-I.R spectrum ( $\text{Cm}^{-1}$ )** ( $\text{NH}_2$ ) str. (3489-3442), (N-H) str. sulfone (3373), (C=N) str. imine (1641), (C=N) str. Pyrimidine(1678), (C-H) str. Pyrimidine(3172), (C-H) str aliphatic (2918-2850), (C=C) str. aromatic (1595-1573), (C- $\text{NO}_2$ )(1516-1338), (N=N)(1419),  $\text{SO}_2$ (1253), (C-O) (1220).

**$^1\text{H}$ NMR spectrum**, ( $\delta$  ppm), (DMSO-*d*<sub>6</sub> MHz), (s, 3H,  $\text{CH}_3$ )( 3.36), (s, 3H,  $\text{OCH}_3$ )( 3.713), (2H,  $\text{NH}_2$ , Aromatic) (s, 6.004), (Ar-H) (m 6.035-- 7.355), (Ar-H) Pyrimidine(m, 7.693-7.916), (NH, sulfon)(s, 10.3).

**$^{13}\text{C}$ -NMR-spectrum**, ( $\delta$  ppm), (DMSO *d*<sub>6</sub>, MHz)(C)( $\text{CH}_3$ ) (24.607), (C)( $\text{OCH}_3$ )(55.945), (C)phenyl rings (121.229, 127.576, 129.544, 129.895), (C)imine(142.375), (C) pyrimidine ring( 105.346, 149.637).

**Synthesis Comp. (II)[4-((Z)-2-(4-aminophenyl)-2-methyl-4,7-dioxo-1,3-oxazepin-3(2H,4H,7H)-yl)-3-(E)-(4-methoxy-2-nitrophenyl)diazenyl)-N-(pyrimidin-2-yl)benzenesulfonamide][15].**

A mixture contain equivalent moles from Schiff base(I)(0.55g, 0.001 mol) and Maleic anhydride(0.098g, 0.001mol) in dry benzene about( 50mL), was refluxed at  $78^{\circ}\text{C}$  for 24 hrs. the reaction was followed by T.L.C. then the mixture was allowed to cool down to room temperature. The resulting solid re-crystallized from Abs. Ethanol. The product is Brown, 68%, m.p. ( $52-54^{\circ}\text{C}$ ) ( $R_f = 0.53$ ) (Met : Tol) (1: 4).

**F.T.I.R spectrum( $\text{cm}^{-1}$ )** ( $\text{NH}_2$ ) str. (3373-3288), (N-H) str. sulfone (3489), (C=O) lacton and (C=O) lactam str. at (1720, 1680), (C=N) str. Pyrimidine(1629), (C-H) str. Pyrimidine(3170), (C=C) oxazepine ring( 1595), (HC=C, oxazepine ring)(3113), (C-H) str aliphatic (2916-2848), (C=C) str. aromatic (1577, 1475), (C- $\text{NO}_2$ )(1516-1338), (N=N)(1421),  $\text{SO}_2$ (1251), (C-O) (1215).

**$^1\text{H}$ NMR spectrum**, ( $\delta$  ppm), (DMSO-*d*<sub>6</sub> MHz) (3H,  $\text{OCH}_3$ )(s 3.8), (3H,  $\text{CH}_3$ )(s 2.086), (s 2H,  $\text{NH}_2$ , Aromatic) (4. 716), (d, 2H,  $\text{CH}=\text{CH}$ , Oxazepine ring) (6.130-6.330), (Ar-H) (m 6.475-- 7.999), (Ar-H) Pyrimidine(m, 8.271-8.996), (NH, sulfon)(s, 11.191).

**C<sup>13</sup>-NMR-spectrum**, ( $\delta$  ppm) ,(DMSO *d*<sub>6</sub>,MHz)(C)(CH<sub>3</sub>) (29.491),(C)(OCH<sub>3</sub>)(55.963-56.532),(C)(N-C-O)<sub>oxazepin</sub> (60.920),(C)phenyl rings (105.328,121.263, 123.717, 127.636,127.995, 129.532, 130.982,131.711, 142.330,142.423, 144.142) (C)(CH=CHcyclic)(120.673,130.125),(C) pyrimidine ring (109.623,149.650), (C)(C=O lactam)(156.990), (C)(C=O lacton)(167.284).

**Synthesis comp.(III)4-((5Z)-2-(4-(4-(dimethylamino)benzylideneamino)phenyl)-2-methyl-4,7-dioxo-1,3-oxazepin-3(2H,4H,7H)-yl)-3-((E)-(4-methoxy-2-nitrophenyl)diazenyl)-N-(pyrimidin-2-yl)benzenesulfonamide [16]:** This Schiff base was prepared from the reaction of comp. (II) (0.644gm ,0.001 mole), with(0.15gm, 0.001mole)of ( *p*-N,N-dimethyl benz aldehyde) in (25ml ) absolute ethanol and (2) drops of glacial acetic acid. This mixture was refluxed for (30)hrs at (80 °C). The progress of the reaction was followed by TLC. After the completion The mixture was cooled down to room temperature then the solid recrystallized from absolute ethanol . T.L.C. (Met:Tol) (1: 4) R<sub>f</sub> = 0.64 , Sienna ,50 % , m.p.(200 °C decomp.).

**F.T.I.R spectrum(cm<sup>-1</sup>)**(N-H) str. sulfone (3427),(C-H) aliphatic str.(2927-2854), (O-C=O) lacton str. (1726), (N-C=O) lactam str. (1691),(C=N)imine group interference with (C=C) oxazepine ring (1577), (C=N) Pyrimidine str. (1660), (C=C) str. aromatic (1533,1498), (C-NO<sub>2</sub>)(1514-1381), (N=N)(1427), SO<sub>2</sub>(1240), (C-O).

**<sup>1</sup>H NMR spectrum**( $\delta$  ppm), (DMSO-*d*<sub>6</sub> MHz),(s 6H, N-(CH<sub>3</sub>)<sub>2</sub>) (3.022), (s 3H,CH<sub>3</sub>)( 1.864), (3 H,OCH<sub>3</sub>)(s 3.854), (d,2H,CH=CH , Alkene) (6.642-6.804), (Ar-H) (m 6.913-7.926), (Ar-H) Pyrimidine(m ,8. 089-8.505) , (1H, CH=N) (s 8.614), (NH, sulfon)(s ,11.039).

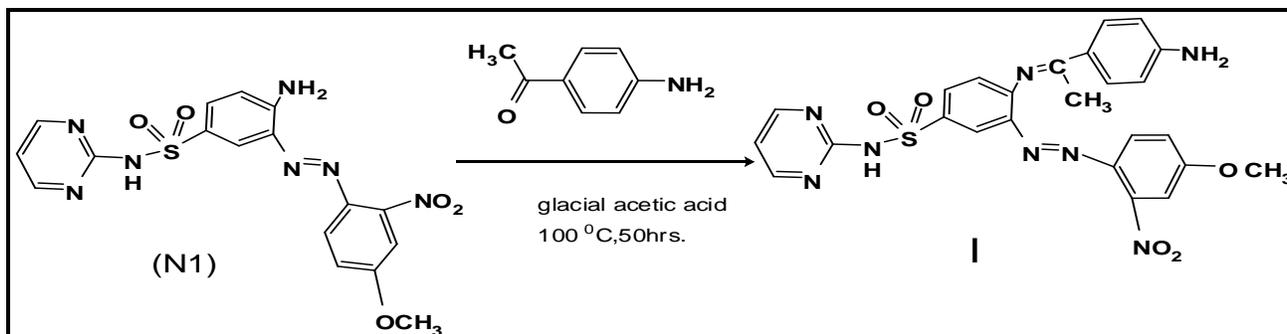
**C<sup>13</sup>-NMR-spectrum**, ( $\delta$  ppm) ,(DMSO *d*<sub>6</sub>,MHz),(C) (CH<sub>3</sub>)(24.647),(C)(N(CH<sub>3</sub>)<sub>2</sub>)(26.865),(C)(OCH<sub>3</sub>)( 55.978).(C)(C=O lactam and lacton) (169.410),(C)(C=C cyclic)(121.273, 129.924) ,(C) imine group (142.426),(C)(N-C-O)<sub>oxazepin</sub>(105.366), (C) pyrimidine ring(149.667,118.624),(C)phenyl rings(127.622,129.566, ,131.988,144.128).

**Synthesis comp.(III)[4-((Z)-2-(4-(3-chloro-2-(4-(dimethylamino)phenyl)-4-oxoazetid-1-yl)phenyl)-2-methyl-4,7-dioxo-1,3-oxazepin-3(2H,4H,7H)-yl)-3-((E)-(4-methoxy-2-nitrophenyl)diazenyl)-N-(pyrimidin-2-yl)benzenesulfonamide][17].**

To a mixture of Schiff bases(III) (0.775gm,0.001 mol) in dioxane (30ml)and Et<sub>3</sub>N (0.35 ml, 0.0025 mol), chloro acetyl chloride (0.2 ml, 0.0025 mol) was added drop-wise at( 5-10 °C). The reaction mixture was stirred for (48 hrs) at room temperature ,then poured into crushed ice to dissolveThe salt(Et<sub>3</sub>N<sup>+</sup> HCl) tri ethyl amine hydrochloride. The mixture was extracted by using chloroform(CHCl<sub>3</sub>) ,then the solvent was evaporated and the yield was re-crystallized from absolute ethanol. the reaction was monitored by (T.L.C).

## RESULTS AND DISCUSSION

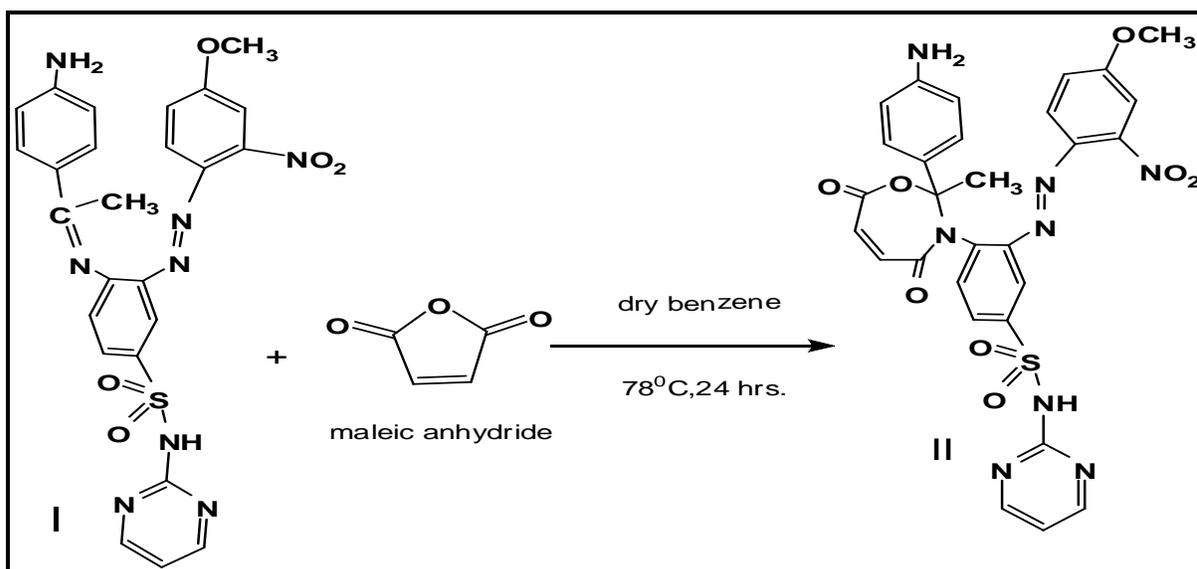
In this study, the derivatives prepared from Azo comp. were identified by comparing their spectra. The derivative(I) was prepared from Azo comp. (N1) with "p-amino acetophenone " via the condensation reaction by using glacial acetic acid as solvent. Here the reaction time about (50 hrs.)is longer than in the case of aldehyde In other words, in the condensation reaction the ketone react more slowly than the aldehyde, due to that the reaction centre of ketone are sterically more hindered than that of aldehyde, also the carbon of ketone less "electrophilic" compared to an aldehyde[18].



**Scheme 1: preparation of Schiff Base (I)**

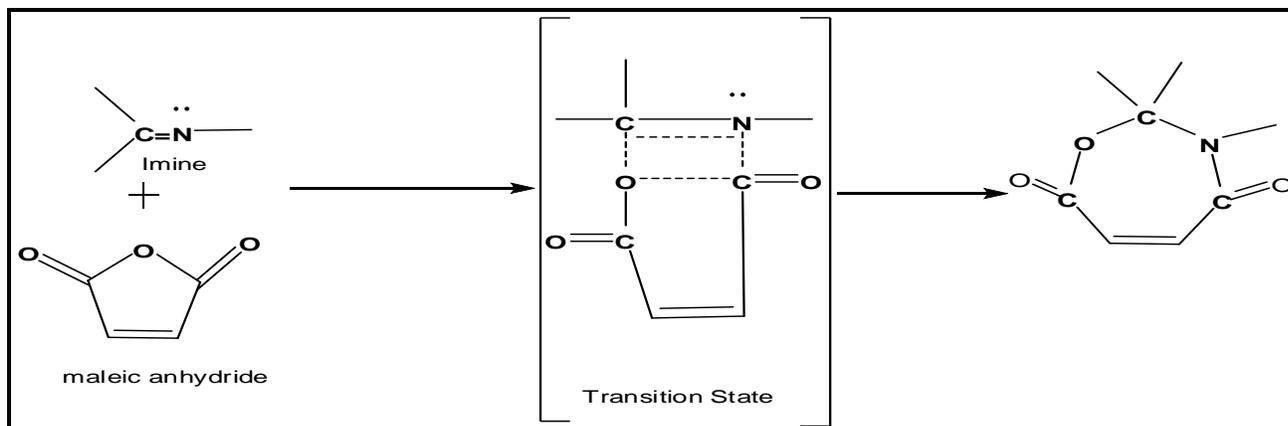
By FT-IR spectrum appearance the absorption band of imine group (C=N) at  $(1641) \text{ cm}^{-1}$ , (C=N) str. Pyrimidine at  $(1678) \text{ cm}^{-1}$  and appearance the low intensity absorption bands than in comp. (N1) attributed to aromatic amine and (NH)sulfon amide at  $(3489-3442)$  &  $(3373) \text{ cm}^{-1}$  respectively. Moreover appearance the stretching absorption of (C-H) aliphatic at  $(2918-2850) \text{ cm}^{-1}$ .  $^1\text{H-NMR}$  spectrum (ppm) (DMSO-*d*<sub>6</sub>) gives (s, 2H, NH<sub>2</sub>) at (6.004), (s, 3H, CH<sub>3</sub>) which linked with imine group at (2.498), (s, 3H, OCH<sub>3</sub>) at (3.713) and (s, 1H, NH) sulfon amide at (10.3).  $^{13}\text{C-NMR}$  spectrum (ppm) (DMSO-*d*<sub>6</sub>) for the Schiff base (I) gives (C) of Methyl group at (24.607), (C) of the Methoxy group at (55.945) and (C) imine group at (142.375).

The derivative (II) was prepared via [2+5] cyclo addition reaction between Schiff base (I) and maleic anhydride in dry benzene as solvent with reflux at  $78^\circ\text{C}$  for (24) hrs.



**Scheme 2: preparation of Comp. (II)**

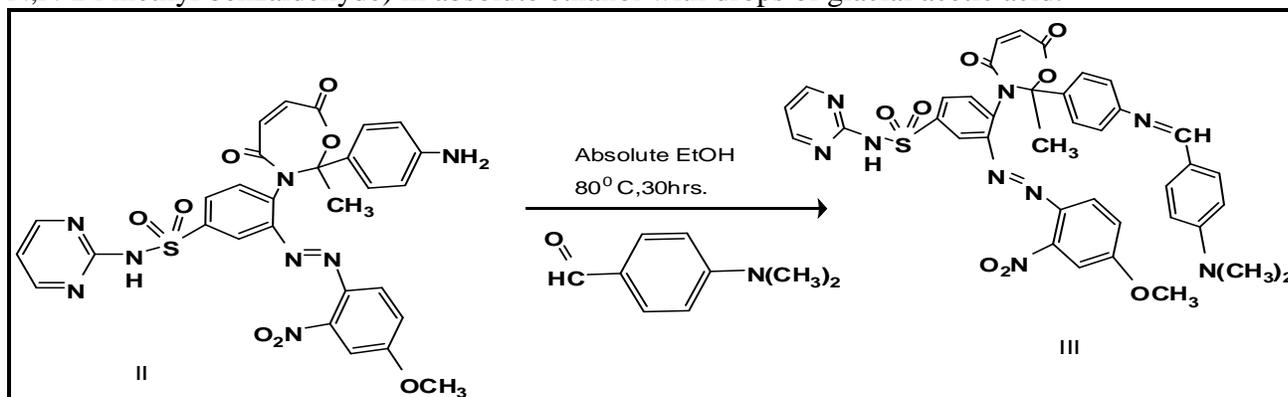
The proposed mechanism<sup>(19)</sup> for the addition of Maleic anhydride to the imine group (C=N) is illustrated in the Scheme(3).



**Scheme(3):Mechanism formation of oxazepine ring**

The derivative identified by FT-IR,  $^1\text{H-NMR}$  and  $^{13}\text{C-NMR}$  techniques. in FT-IR spectrum observed appearance the stretching vibration of lactone and lactam for Oxazepine ring at  $(1720,1680)\text{ cm}^{-1}$  respectively and the two stretching bands of aromatic amine at lower wave number in  $(3373-3288)\text{ cm}^{-1}$  while the band of (NH)sulfonamide appeared at higher wave number in  $(3489)\text{ cm}^{-1}$ . (C=C) olefinic of oxazepine ring at  $(1595)\text{ cm}^{-1}$ .  $^1\text{H-NMR}$  spectrum (ppm)(DMSO-*d*<sub>6</sub>) shows the signals at  $(2.066), (3.8)$  and  $(4.716)$  due to (s,3H,CH<sub>3</sub>) linked with oxazepine ring, (s,3H,OCH<sub>3</sub>) and (s,2H,NH<sub>2</sub>) respectively. The olefinic protons of oxazepine ring showed at  $(6.130-6.330)$  and (s,1H,NH-sulfonamide) at  $(11.191)$ .  $^{13}\text{C-NMR}$  spectrum (ppm)(DMSO-*d*<sub>6</sub>) gives signals at  $(29.491,55.963-56.532, 60.920)$  due to (C) atoms of (CH<sub>3</sub>) Associated with oxazepine ring, (OCH<sub>3</sub>) and (N-C-O) oxazepine ring respectively. (C) of carbonyl lactone and lactam Oxazepine ring at  $(167.284,156.990)$ . The signal of carbonyl lactone is higher value because of link it with (O) atom for Oxazepine ring. (C=C cyclic)  $(130.125)$  for (C) close to lactone,  $(120.673)$  for (C) close to lactam<sup>(20,21)</sup>.

The Schiff base(III) was prepared through condensation reaction between the derivative (II) and (p-N,N-Di methyl benzaldehyde) in absolute ethanol with drops of glacial acetic acid.

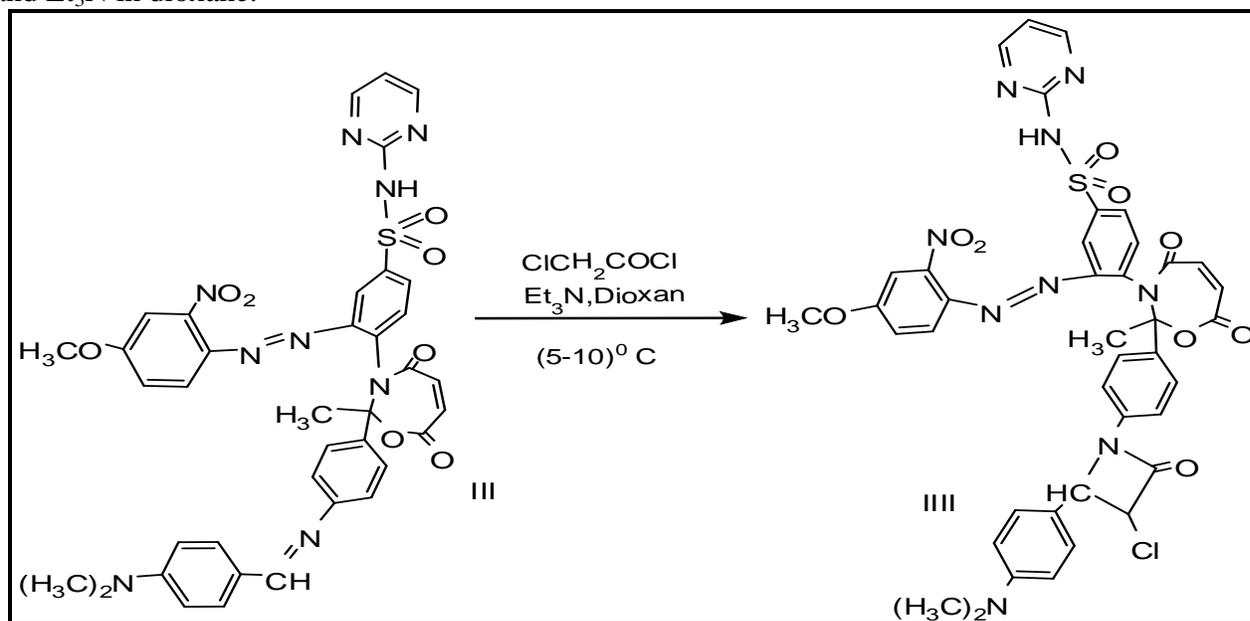


**Scheme 4: preparation of Comp. (III)**

The derivative identified by FT-IR spectrum through disappearance the two vibration bands of amine group and appearance the vibration band for imine group interference with band of (C=C Oxazepine) olefinic at  $(1577)\text{ cm}^{-1}$  because of many functional groups in same position from spectrum, expecting that it appeared under other bands which in same position such as (C=N) pyrimidine or (C=C Oxazepine) due to crowding of functional groups in this area. in addition to appearance shoulder band due to the lactone oxazepine ring at  $(1726)\text{ cm}^{-1}$  while the lactam

appeared at ( 1691 )  $\text{cm}^{-1}$ . the band at(1660)  $\text{cm}^{-1}$  belong to (C=N)pyrimidine also the band of (NH)sulfon amide was appeared at (3427)  $\text{cm}^{-1}$ .  $^1\text{H-NMR}$  spectrum(ppm)(DMSO-*d*6) shows (s,6H,N-( $\text{CH}_3$ )<sub>2</sub>) at(3.022), (s, 3H,  $\text{CH}_3$ .) linked with Oxazepine ring at(1.864), (s,3 H , $\text{OCH}_3$ ) at(3.854) , (CH=CH olifinic cyclic ) at(6.642-6.804) and (s,1H ,NH- sulfonamide) at( 11.039).  $^{13}\text{C-NMR}$  spectrum(ppm)(DMSO-*d*6) gives (C)atoms for ( $\text{CH}_3$ ),(N( $\text{CH}_3$ )<sub>2</sub>) and ( $\text{OCH}_3$ )at(24.647, 26.865and 55.978) respectively, (C) carbonyl lacton and lactam at (169.410), (C=C cyclic)at (121.273, 129.924) and (C) (C=N) group at(142.426).

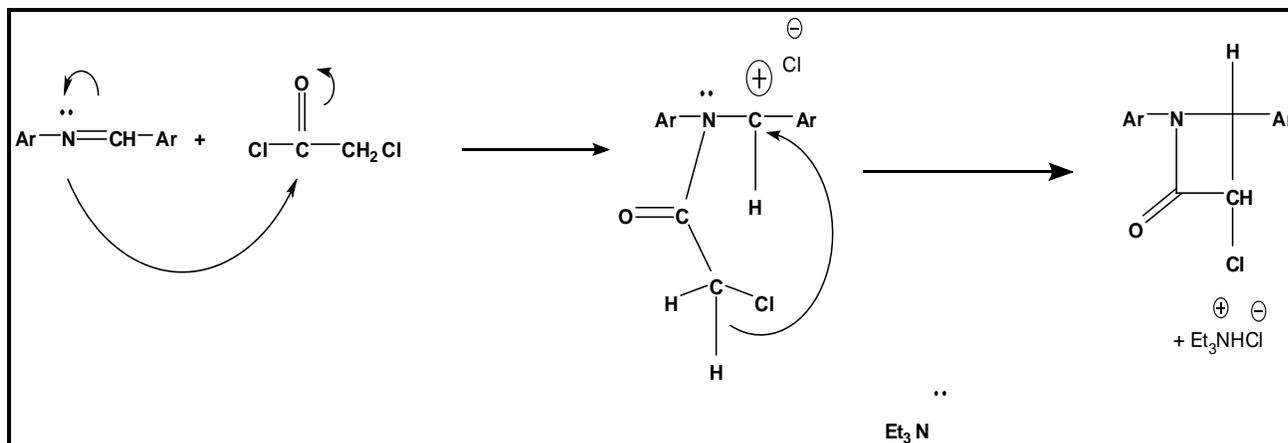
The derivative (III)was prepared by[2+2] cycloaddition reaction from (II),chloro acetyl chloride and  $\text{Et}_3\text{N}$  in dioxane.



#### Scheme 5: preparation of Comp. (III)

The chemical structure of (III) was confirmed through FT-IR spectrum by appearance the stretching vibration of carbonyl lactam (four membered ring ) and carbonyl lacton of oxazepine ring at high wave number (1757)  $\text{cm}^{-1}$ , while the carbonyl lactam in oxazepine ring appeared at (1691)  $\text{cm}^{-1}$ , the band at(1670)  $\text{cm}^{-1}$  belong to (C=N)pyrimidine. the band of (C=C Oxazepine) olefinic at(1620)  $\text{cm}^{-1}$  also the band of (NH)sulfon amide was appeared at (3408)  $\text{cm}^{-1}$ .  $^1\text{H-NMR}$  spectrum(ppm)(DMSO-*d*6) figure(3-50) gives (s,6H,N-( $\text{CH}_3$ )<sub>2</sub>) at(3.523) , (s, 3H, $\text{CH}_3$ ) at(1.255), (s,3H, $\text{OCH}_3$ ) at(3.865), (d,1H ,N-CH-) at (4.340) ,small (d,1H ,O=C-CH-Cl)at about(4.8), (HC=CH cyclic)olefinic small signals at about(6.8) and (s,1H ,NH- sulfonamide)at (10.494).

The mechanism<sup>(14)</sup> of [2+2] cyclo addition to prepare mono- $\beta$ -lactam shown in the Scheme(6).



**Table( 1) some physical properties of prepared derivatives**

Comp.	m.p.°C	Yield%	Color	M .Wt	M.F	Rf of T.L.C.
I	80-84	67	Dark red	546	C <sub>25</sub> H <sub>22</sub> N <sub>8</sub> O <sub>5</sub> S	0.52 (*Met: **Tol)2:3
II	52-54	68	Brown	644	C <sub>29</sub> H <sub>24</sub> N <sub>8</sub> O <sub>8</sub> S	0.53  (Met: Tol)1:4
III	200 decomp.	50	Sienna	775	C <sub>38</sub> H <sub>33</sub> N <sub>9</sub> O <sub>8</sub> S	0.64  (Met: Tol)1:4
III	280 decomp	49	Brown	851.5	C <sub>40</sub> H <sub>34</sub> ClN <sub>9</sub> O <sub>9</sub> S	0.54 (Met: Tol)1:4

\* Methanol , \*\*Toluene

**Table( 2) FT-IR bands of prepared derivatives**

Comp.	FT-IR(cm <sup>-1</sup> )
I	( NH <sub>2</sub> )str. (3489-3442), (N-H) str. sulfone (3373), (C=N) str.imine (1641), (C=N) str. Pyrimidine(1678), (C-H) str . Pyrimidine(3172), (C-H) str aliphatic (2918-2850),(C=C) str aromatic (1595-1573), (C-NO <sub>2</sub> )(1516-1338),(N=N)(1419), SO <sub>2</sub> (1253), (C-O) (1220)

II	(NH <sub>2</sub> )str. (3373-3288), (N-H) str. sulfone (3489), (C=O) lacton and (C=O) lactam str. at (1720, 1680), (C=N) str. Pyrimidine(1629), (C-H) str. Pyrimidine(3170), (C=C) oxazepine ring( 1595),(HC=C, oxazepine ring)(3113),(C-H) str aliphatic (2916-2848),(C=C) str. aromatic (1577,1475), (C-NO <sub>2</sub> )(1516-1338), (N=N)(1421), SO <sub>2</sub> (1251), (C-O) (1215).
III	(N-H) str. sulfone (3427),(C-H) aliphatic str.(2927-2854), (O-C=O) lacton str. (1726), (N-C=O) lactam str. (1691),(C=N)imine group interference with (C=C) oxazepine ring (1577), (C=N) Pyrimidine str. (1660), (C=C) str. aromatic (1533,1498), (C-NO <sub>2</sub> )(1514-1381), (N=N)(1427), SO <sub>2</sub> (1240), (C-O) (1180).
III	(N-H) str. sulfone (3408),(C-H) aliphatic str.(2972-2926), (C=O) str. (1757) including (β-lactam ring and lacton oxazepine), lactam oxazepine ring (1691), (C=C)str. oxazepinering(1620),(C=N)str.pyrimine ring(1670),(C=C) str. aromatic(1593),(C-NO <sub>2</sub> )(1519-1354), (N=N)(1458-1444), (C-Cl)(881).

Table( 3) <sup>1</sup>H-NMR Signals of prepared derivatives

Comp.	<sup>1</sup> H-NMR(ppm)
I	( s,3H, CH <sub>3</sub> )( 3.36), ,( s,3H,OCH <sub>3</sub> )( 3.713), (2H,NH <sub>2</sub> ,Aromatic) ( s, 6.004), (Ar-H) (m 6.035-- 7.355), (Ar-H) Pyrimidine(m ,7.693-7.916) , (NH, sulfon)(s ,10.3)
II	(3H,OCH <sub>3</sub> )(s 3.8),( 3H,CH <sub>3</sub> )(s 2.086), ( s 2H,NH <sub>2</sub> ,Aromatic) (4. 716), (d,2H,CH=CH , Oxazepine ring) (6.130-6.330), (Ar-H) (m 6.475-- 7.999), (Ar-H) Pyrimidine(m ,8.271-8.996) , (NH, sulfon)(s ,11.191).
III	( s 6H, N-(CH <sub>3</sub> ) <sub>2</sub> ) (3.022), ( s 3H,CH <sub>3</sub> )( 1.864), (3 H,OCH <sub>3</sub> )(s 3.854), ( d,2H,CH=CH , Alkene) (6.642-6.804), (Ar-H) (m 6.913-7.926), (Ar-H) Pyrimidine(m ,8. 089-8.505) , (1H, CH=N) ( s 8.614), (NH, sulfon)(s ,11.039)
III	(6H, N-(CH <sub>3</sub> ) <sub>2</sub> ) (s 3.523), ( 3H,CH <sub>3</sub> )(s 1.255), (3 H,OCH <sub>3</sub> )(s3.865),(1H,N-CH-lactam)(d4.340),(1H,O=C-CH-Cl <sub>lactam</sub> )(d,4.8), small signals about(6.8)(HC=CH cyclic)olifinic.(1H)(NH sulfon )(s,10.494),(Ar-H) (m 7.339-7.948), (NH, sulfon)(s ,11.039)

Table( 4) <sup>13</sup>C-NMR Signals of prepared derivatives

Comp.	C-NMR(ppm) <sup>13</sup>
I	(C)(CH <sub>3</sub> ) (24.607), (C)(OCH <sub>3</sub> )(55.945), (C)phenyl rings (121.229, 127.576,129.544,129.895) (C)imine(142.375),(C) pyrimidine ring( 105.346, 149.637).
II	(C)(CH <sub>3</sub> ) (29.491),(C)(OCH <sub>3</sub> )(55.963-56.532),(C)(N-C-O) <sub>oxazepin</sub> (60.920),(C)phenyl rings (105.328,121.263, 123.717, 127.636,127.995, 129.532, 130.982,131.711, 142.330,142.423, 144.142) (C)(CH=CHcyclic)(120.673,130.125),(C) pyrimidine ring(109.623,149.650), (C)(C=O lactam)(156.990), (C)(C=O lacton)(167.284).
III	(C) (CH <sub>3</sub> )(24.647),(C)(N(CH <sub>3</sub> ) <sub>2</sub> )(26.865),(C)(OCH <sub>3</sub> )( 55.978),(C)(C=O lactam and lacton) (169.410),(C)(C=C cyclic)(121.273, 129.924) ,(C) imine group (142.426),(C)(N-C- O) <sub>oxazepin</sub> (105.366), (C) pyrimidine ring(149.667,118.624),(C)phenyl rings(127.622,129.566 ,131.988,144.128).

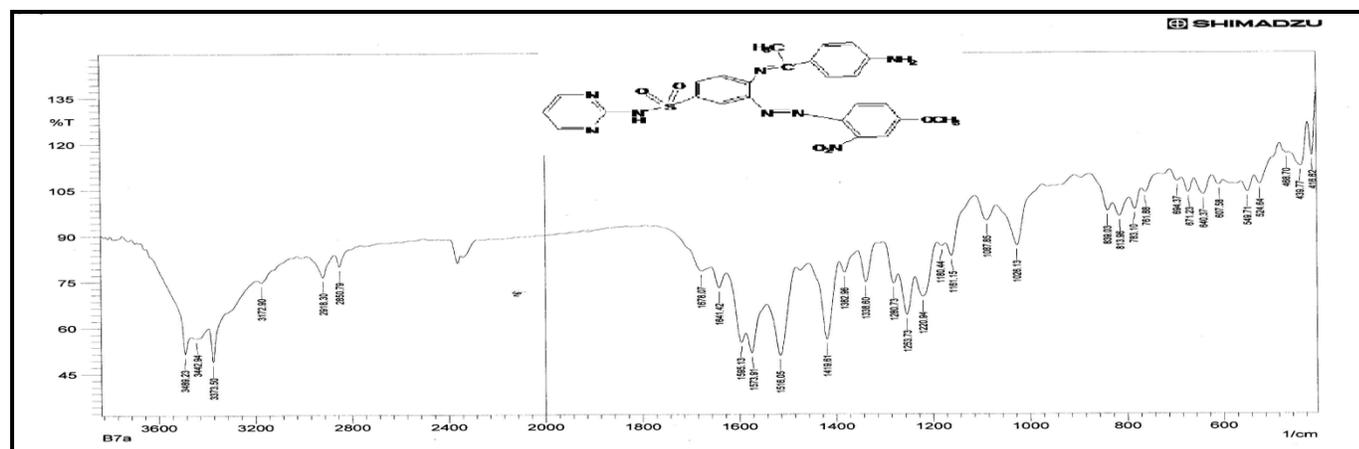


Figure (1) FT-IR spectrum of Schiff base(I)

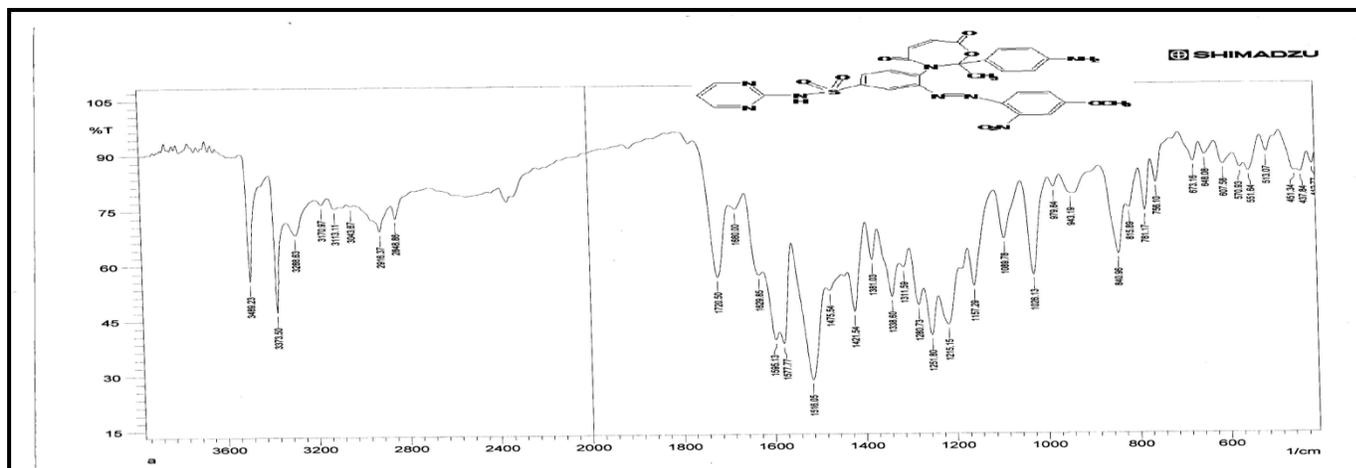


Figure (2) FT-IR spectrum of Oxazepine ring derivative(II)

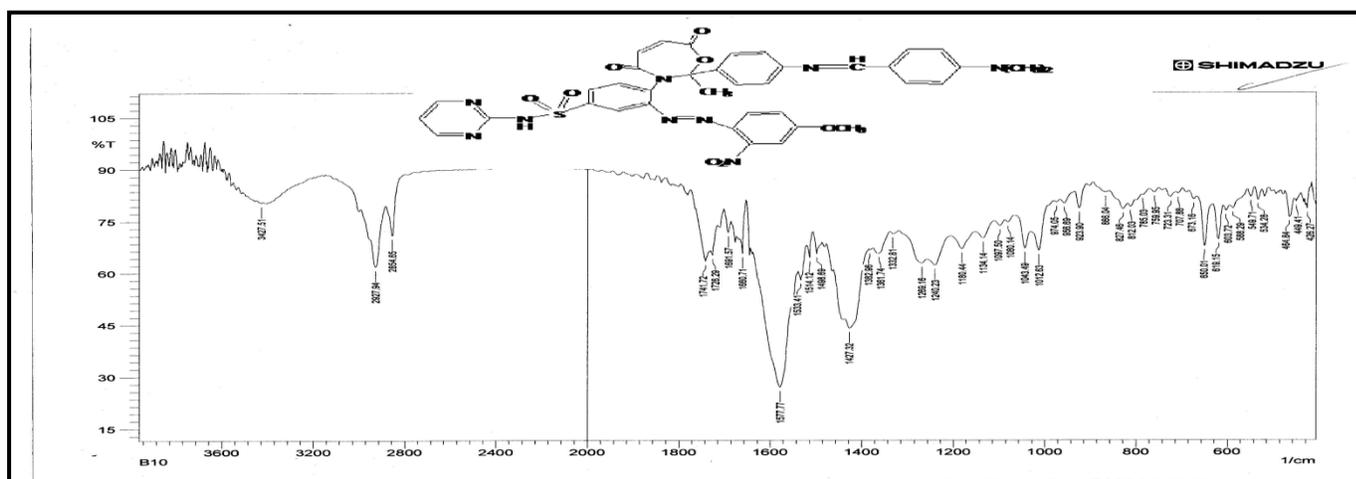


Figure (3) FT-IR spectrum of Schiff base(III)

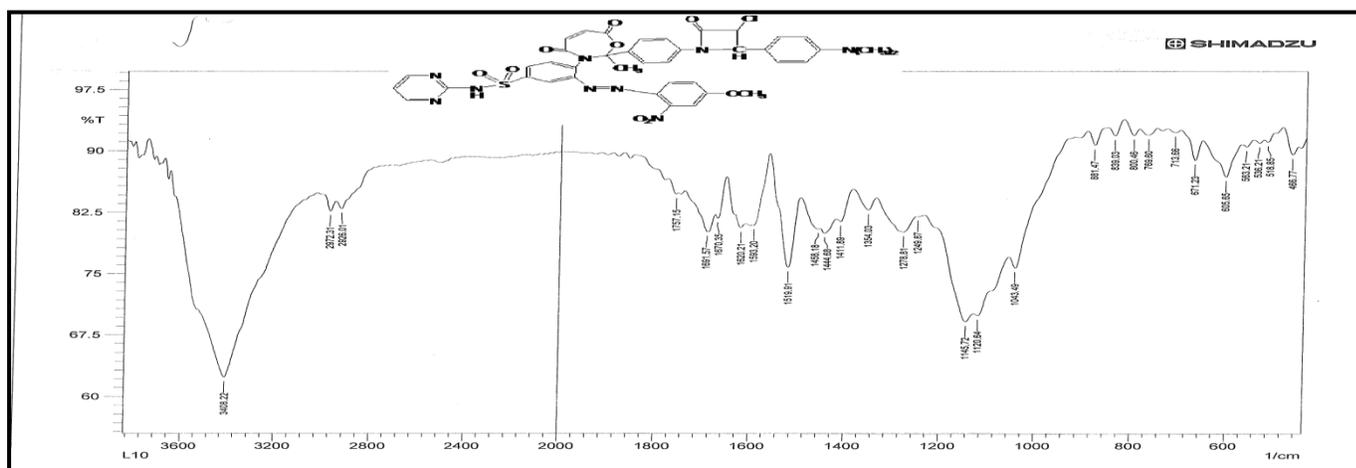


Figure (4) FT-IR spectrum of Lactam ring derivative(III)

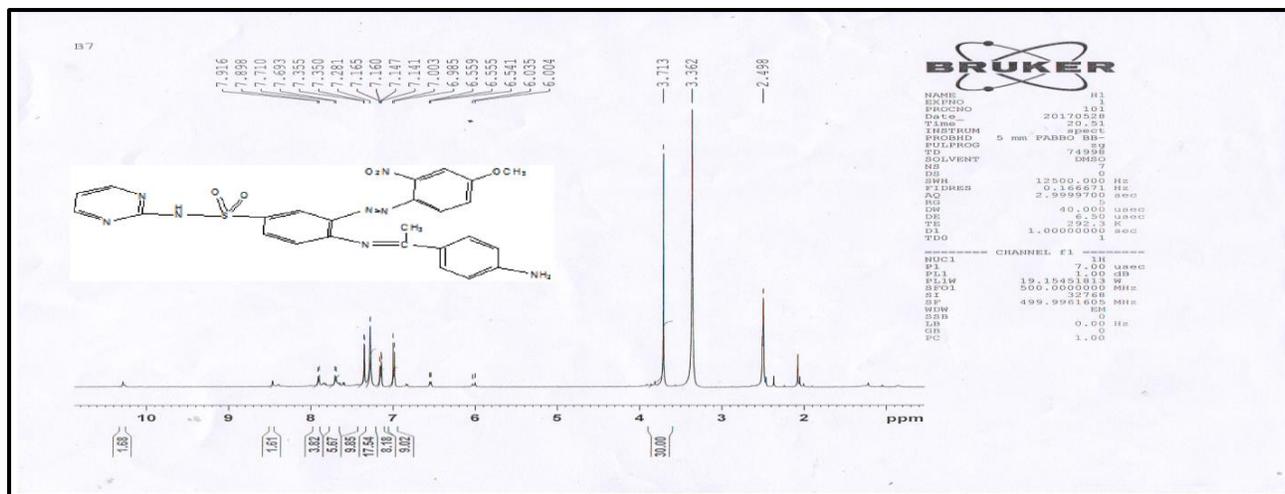


Figure (5) <sup>1</sup>H-NMR spectrum of Schiff base(I)

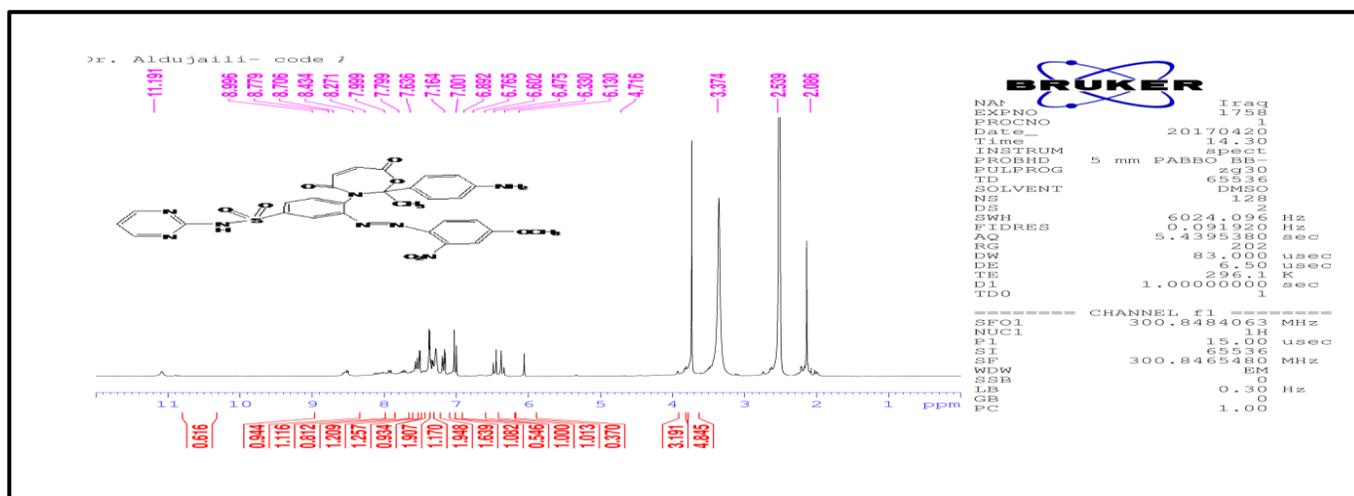


Figure (6) <sup>1</sup>H-NMR spectrum of Oxazepine ring derivative(II)

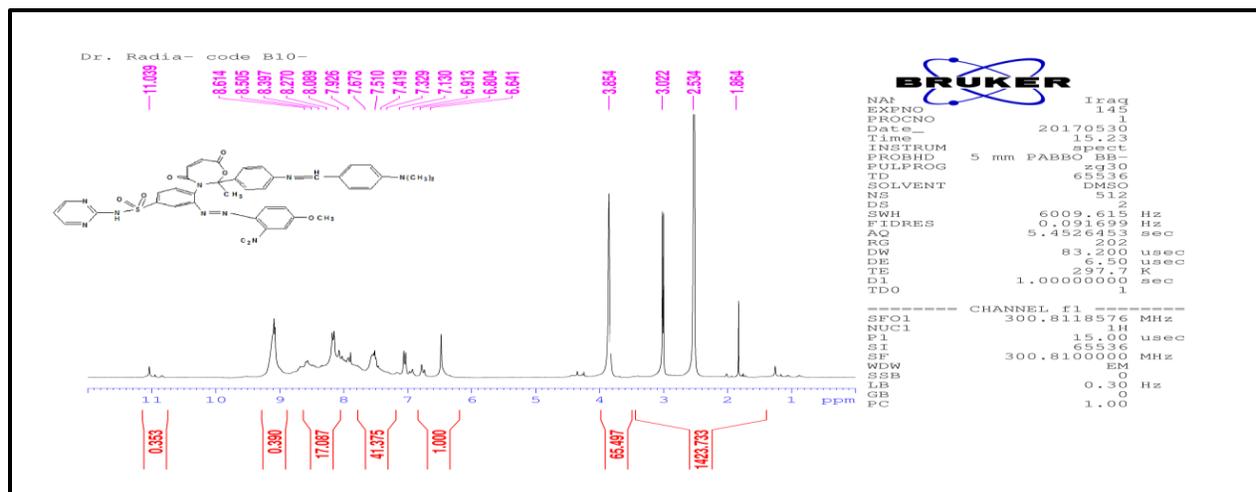




Figure (9)  $^{13}\text{C}$ -NMR spectrum of Oxazepine ring derivative(II)

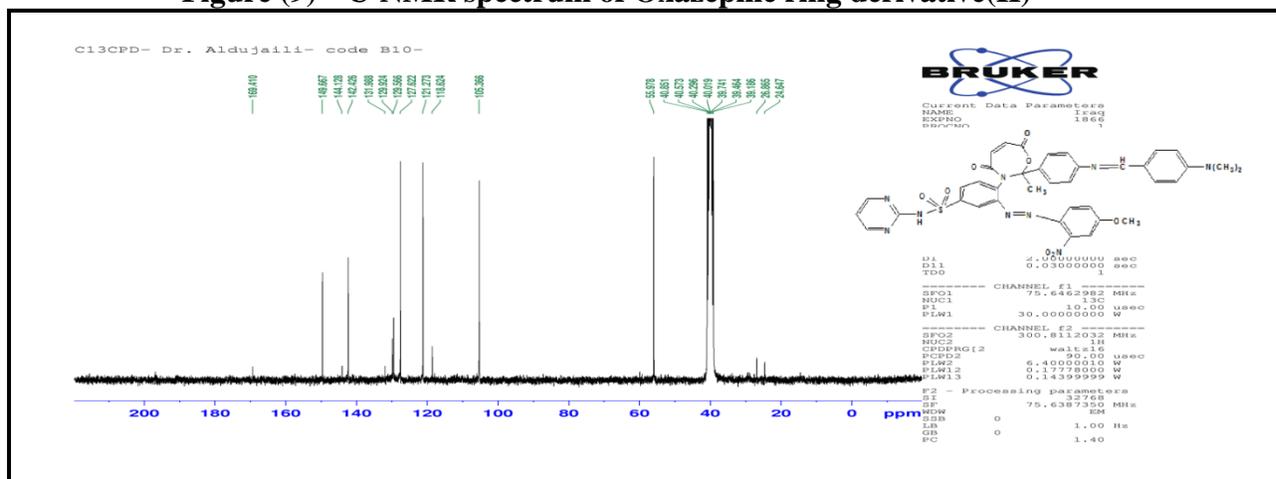


Figure (10)  $^{13}\text{C}$ -NMR spectrum of Schiff base(III)

## CONCLUSION

In this study we are reported synthesis of many  $\beta$ -Lactam derivatives via Staudinger Reaction[2+2]cyclo addition. The work included preparation of Azo-Schiff base compounds from sulphadiazine as the first step then cyclization process for these compounds by using chloro acetyl chloride in the basic medium and low temp. (0-10) °C These derivatives were found to be stable at room temperature .These derivatives confirmed from spectral data analysis; FT-IR , $^1\text{H}$ NMR and $^{13}\text{C}$ NMR.

## REFERENCES

- 1-Jiaxi Xu, "Stereo selectivity in the synthesis of 2-azetidinones from ketenes and imines via the Staudinger reaction". [2009]. *ARKIVOCix* Pp. 21-44.
- 2-Singh, G.S., Recent progress in the synthesis and chemistry of azetidinones. [2003]. *Tetrahedron* 59: 7631–7649.
- 3- Radhiyah A. Khdur and Ezzat H. Zimam, Synthesis, Characterization And Study Biological Screening Of Some New Azetidinone Derivatives From Azo- Sulphadiazine. [2018]. *Pak. J. Biotechnol.* Vol. 15 (1) 201-217.
- 4-A. Salim Mohammed and Y. S. HaseebZebary;"Spectrophotometric Determination of Sulfadiazine via Diazotization and Coupling Reaction - Application to Pharmaceutical Preparations",[2013]. *Raf. J. Sci*, 24(6), pp. 61-73,.
- 5-Who Model List of Essential Medicines;" *World Health Organization*. October (2013), Retrieved 22 April (2014).
- 6-A. A. Mukhlus , M. S. Al-Rawi, J. H. Tomma, A. H. Al-Dujaili,," Synthesis And Characterization Of New Oxazepines Derived From D-Erythroascorbic Acid ",[2012]. *Ibn Al-Haitham Journal for Pure and Applied Science*, 25 (2).
- 7-Dhanya Sunil , Ranjitha C , Rama M , KSR Pai," Oxazepine Derivative as an Antitumor Agent and Snail Inhibitor against Human Colorectal Adenocarcinoma ", August [2014]. *International Journal of Innovative Research in Science, Engineering and Technology*, 3( 8).

- 8-Fred. van der Steen and Gerard van Koten , "Syntheses of 3-Amino-2-azetidinones: A Literature Survey", [1991]. *Tetrahidron* 41( 36), Pp. 7503-7524.
- 9-R. Deshmukh , A. Kumar Jha , A. Singh Thakur and P.SudhirKumar., "Synthesis And Biological Activity Of Some Thiazole Compounds Containing Azetidinone And Thiazolidinones Derivatives ", [2014]. *World Journal of Pharmaceutical research*, 3( 2),.
- 10-A.A.Ashokrao, "Synthesis And Biological Activities Of Some Substituted Azetidinones" (dissertation), (2006 ) Rajiv Gandhi University of health sciences, Karnataka, 23.
- 11-Block JH, Beale JM, [2004]: Wilson and Gisvolds textbook of Organic Medicinal and Pharmaceutical Chemistry, 11<sup>th</sup> edition; Lippincott Company, 299.
- 12- Faikong K., Schneper L. and Mathee K., "Beta-lactam Antibiotics: from Antibiosis to Resistance and Bacteriology", [2009] . *The Authors Journal Compilation APMIS*, 1-36.
- 13-Muthanna D. Saud, Muna .I. Khalaf, Huda.A.Hassan and Ikbal .R.Hanna., "Synthesis of New Derivatives of  $\beta$  - Lactam Antibiotics", [ 2014]. *International Journal of Pharm Tech Research*, 6(3), pp 1018-1027.
- 14-Radiyah A. KhdurR and Ezzat H. Zimam, " Synthesis and Characterization of some new  $\beta$ -Lactam Derivatives from Azo Sulphadiazine Compound and Study Biological Evaluation as Anticancer for one of them", [2018]. *Oriental Journal Of Chemistry*, Vol. 34, No.(1): Pg. 371-380.
- 15-A. AQ .Younus and N. R .Jber , "Synthesis and Characterization a New 1,3-Oxazepine Compounds from New Bis-4-Amino-3-mercapto-1,2,4-triazole Derivatives", June [2016]. *Organic Chemistry: An Indian Journal*, 12 ( 2 ) .
- 16- Hassan.T.Ghanem and Radiyah.A.Al-dujaili, "Synthesis new heterocyclic derivatives from Schiff base 2-Amino(2-imine furan)", [2013]. *AL-taqani Journal*, 26(2).
- 17-Entesar O. AlTamiemi, Sameaa J. Khammas and Sura S. AlKaissi, " Synthesis, Characterization and Study the Biological Activity of New Morpholine Derivative", [2015]. *Baghdad Science Journal*, 12(4).
- 18-161-John. McMurry ., "Organic chemistry" seventh Edition, THOMSON, BROOKS/ COLE, (2008).
- 19- Nagham. M. AL-Jamali. [2008]. Chemistry Department , College of Education , University of Baghdad. **ph. D. Thesis**.
- 20-Sh.Adnan, A. J. Mohammed and H. Thamer., "Synthesis and identification of some derivatives of 1,3,4-thiadiazole", [2015]. *Journal of Chemical and Pharmaceutical Research*, 7(10): 1000-1011,.
- 21-Kh. M. Mohammad , M. R. Ahmed and M. H. Mahmoud , "Synthesis and characterization of some new (1,3-Oxazepine) derivative from 6-methyl 2-thiouracil and study their biological activity", [2017]. *Tikrit Journal of Pure Science* , 22 (2).

