



Research Article

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## Synthesis and characterization of $\beta$ -Lactam compounds

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### ABSTRACT

This article involve synthesized of  $\beta$ -lactam compounds from amine derivatives .The first step prepared of azo compounds from 4,4-diamino diphenylsulphon (DDS) with 3-methoxy-4-hydroxybenzaldehyde (Vaniline) .Then prepared of Schiff base from azo compound with 4-aminoantipyrine. This compounds ware characterized by melting point, FT.IR,  $^1\text{H}$  NMR and  $\text{C}^{13}$  NMR.

**Key words:** Heterocyclic compound, Schiff base, Azo dyes,  $\beta$ -lactam derivative.

### INTRODUCTION

Heterocyclic compounds can define as organic compounds which has hetro atom on own structure for example (N, O, S, P, Se, .... Etc. ) [1].

$\beta$ -lactam is a part of four member ring saturated heterocyclic compound containing one nitrogen atom as well as carbonyl group [2] .Its non-aromatic heterocyclic compound [3].its class for Azetidine ring [4] .

most important application of  $\beta$ -lactam in the synthesis of penicillin [5],where its function group for penicillin hence its binding to the active sit of Transpeptidase enzyme which can define as a bacterial enzyme that cross-links the peptidoglycan chains to form rigid cell walls [6-7].

Azo compounds are compounds containing the functional group(R-N=N-R) where R and R' can be alkyl or aryl group , Functional group N = N called azo group [8]. Azo dyes aromatic, it is organic dyes of the most prevalent and the importance considered depending on the one of applications in the textile, medicine [9]. Azo dyes, which are derived from aromatic compounds by replacing the secretary substitutes group on the ring, are of very importance in medical applications and dyes [10]. Also it is considered one of the most important vehicles that have been entered in the industrial field and drug, in addition to being used as an antibiotic [11].

### EXPERIMENTAL SECTION

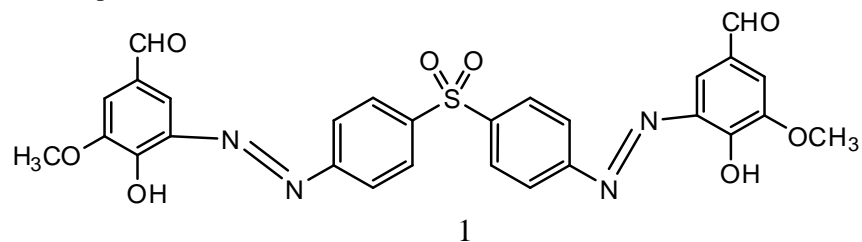
#### Materials

Chemicals used during the current work are the 4,4-diamino diphenylsulphon (DDS), 3-methoxy-4-hydroxybenzaldehyde( Vaniline ),4-aminoantipyrine and was one of the production company Sigma and Aldrich.,in addition to Use of Ethanol and Dioxan as solvents during the process of reflex

#### Instrumentation

Recorded melting point by hot stage Gallen Kamp. To ensure the purity of the resulting compounds used technique Thin layer chromatography (TLC)was carried out, the presence of iodine as an aspect of the spot. F.T.I.R spectroscopy was used KBr disc.  $^1\text{H}$  NMR &  $\text{C}^{13}$  NMR spectra was used ( $\text{CDCl}_3$ ).

1-Preparation of azo compounds [12].



This compound was prepared in two steps

#### The first step:

Formation diazonium salt; 4,4-Diaminodiphenylsulfone (DDS) (0.001 mol) was dissolved in hydrochloric acid (3 ml) and (10 ml) both of water and ethanol, then cooled in ice bath (0-5°C). Sodium nitrite (0.002 mol) was dissolved in (10 ml) distilled water and cooled in ice bath (0-5°C), then slowly mix solution together. After that; at same temperature, the mixture was kept for 20 min.

#### The second step:

Azo dye composition; The 3-methoxy-4-hydroxybenzaldehyde (0.002 mol), was dissolved in sodium hydroxide solution (15 mL, 10%) stirred the solution in the first step at (0-5°C) then added slowly to that solution with stirring, leaving the reaction mixture for a period 3 hrs. Then filtered, and collected the solid product.

Physical properties of compounds are shown in table-1-

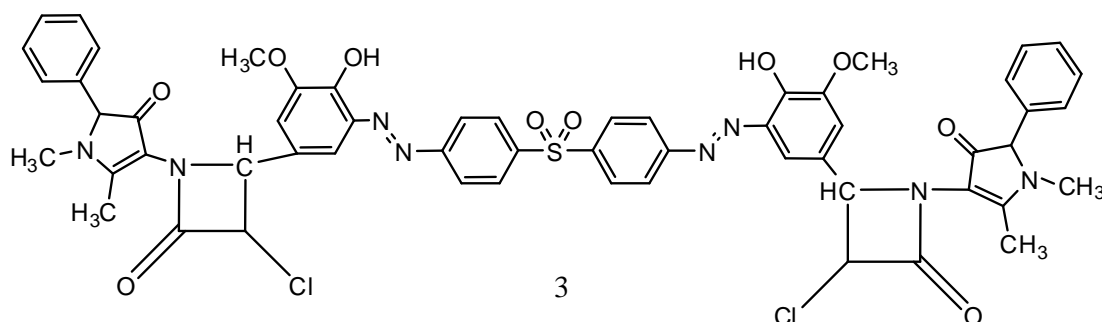
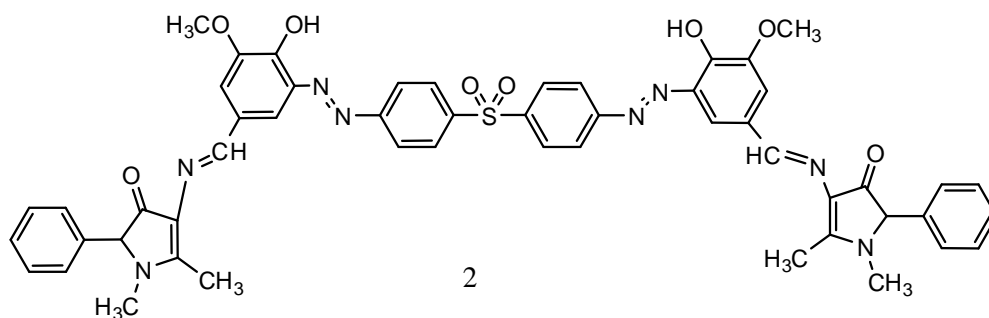


Table (1): Physical properties and other characteristics for the synthesis compounds (1-3)

No.	Molecular Formula	M.Wt g/mol	M.P	Color	Rf	Solvent	Time
1	C <sub>28</sub> H <sub>22</sub> N <sub>4</sub> O <sub>8</sub> S	574.56	189-191	Black	—	EtOH&H <sub>2</sub> O	3 hrs
2	C <sub>52</sub> H <sub>46</sub> N <sub>8</sub> O <sub>8</sub> S	943.04	269-271	Black	0.3	Abs. EtOH	6.30 hrs
3	C <sub>56</sub> H <sub>48</sub> Cl <sub>2</sub> N <sub>8</sub> O <sub>10</sub> S	1096	294-296	Brown	0.4	Dioxan	10 hrs

General procedure for Schiff base from azo dye [13].

Azo compound(1mol)reaction with Amine derivative(4-aminoantipyrine)was add to solution in EtOH absolute(20 mL),and then a catalytic amount of GAA (two or three drops)was mixture at temperature 70C.The reaction mixture was stirred for 3h. The products were collected by filtration and recrystallized by ethanol.

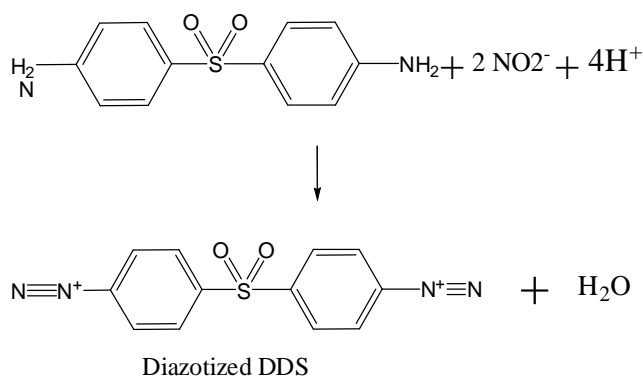
General procedure Synthesis of  $\beta$ -lactam from reaction of Schiff Base with Chloroacetylchloride [14].

The Schiff base (2) (1 mol) reaction with (2 mol) of chloroacetylchloride in (15 ml) Dioxan solution. And then catalyst the interaction with adding drops of triethylamine (2mol). Leads to the formation  $\beta$ -Lactam derivatives

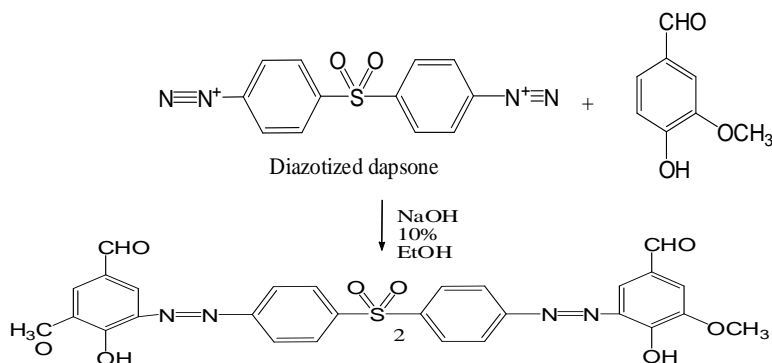
## RESULTS AND DISCUSSION

### Synthesis of azo compound

4,4 -Diaminodiphenylsulfone (DDS) ,in acidic medium ,is react with nitrite to form the diazonium salt:



After around twenty minutes; diazonium salt formation is coupled with 3-methoxy-4-hydroxy- benzaldehyd (Vaniline) in a basic medium ( NaOH 10%)at 0-5C<sup>0</sup> to form Azo dyes (dark color) :



Scheme (1) preparation of Azo dye

The FT-IR spectra of azo compound(1) fig(1),show of(CHO)aldehyde group at(1691.57) $\text{cm}^{-1}$  and(–CH )aromatic band at(3091.89) $\text{cm}^{-1}$ and(CH)aliphatic bands at(2962.66) $\text{cm}^{-1}$ .And at(3458.37) $\text{cm}^{-1}$  absorption(OH)phenolic group, with disappearance the stretching vibration of(NH<sub>2</sub>)group at (3290&3100 $\text{cm}^{-1}$ ) (symmetric & asymmetric)respectively return to (DDS compound).

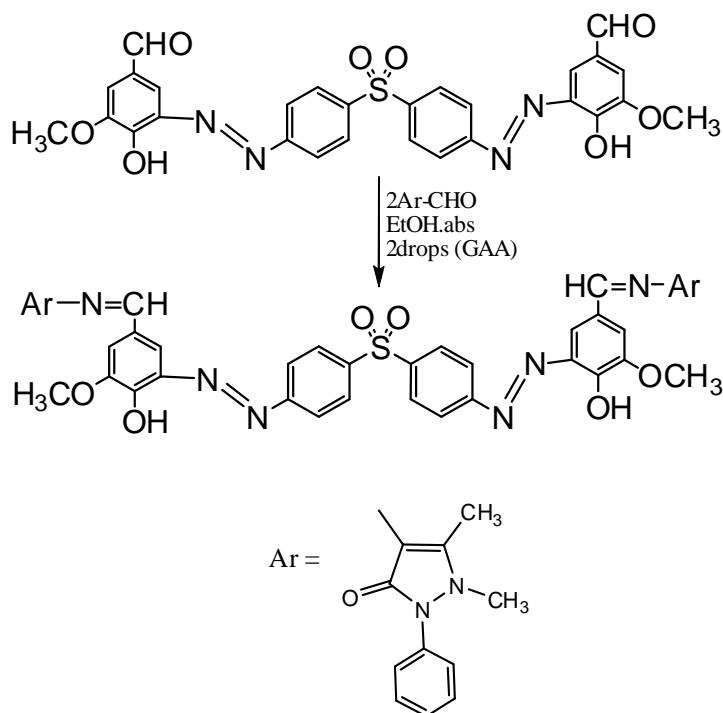
<sup>1</sup>H NMR spectrum fig(4)shows the following characteristic chemical shifts for compound(1),(CDCl<sub>3</sub>)solvent at(2.5)ppm, the proton (OH) appeared at (13.7)ppm, and proton of aliphatic group absorbed at(4.01)ppm, At(7.9-8.1)ppm for aromatic ring proton, and proton of aldehyde group(CHO)appeared at(9.9)ppm.

<sup>13</sup>C-NMR spectrum fig(7)of compound(1)showed the signal in(56.5) ppm for the carbon in methoxy group(OCH<sub>3</sub>).The signal at (111-136)ppm due to the carbon(C=C for aromatic ring).at(152)ppm for the carbon of (C-OH),and at(189.676)ppm for the carbon of carbonyl group.

The suggested mechanism of these compounds in scheme (1) .

**Synthesis of Schiff base compound**

The reaction between Azo compound (1)[Which contain aldehyde group]with 4-aminoantipyrine to synthesize Schiff base(2).



**Scheme (2) preparation of Schiff base**

In the FT-IR spectrum there are three major peaks; which are depending upon the different substitution groups appeared in the compound, and its relative to azomethen groups( $\text{N}=\text{CH}-$ ),( $=\text{CH Ar}$ ) and ( $\text{C-OH}$ )group.

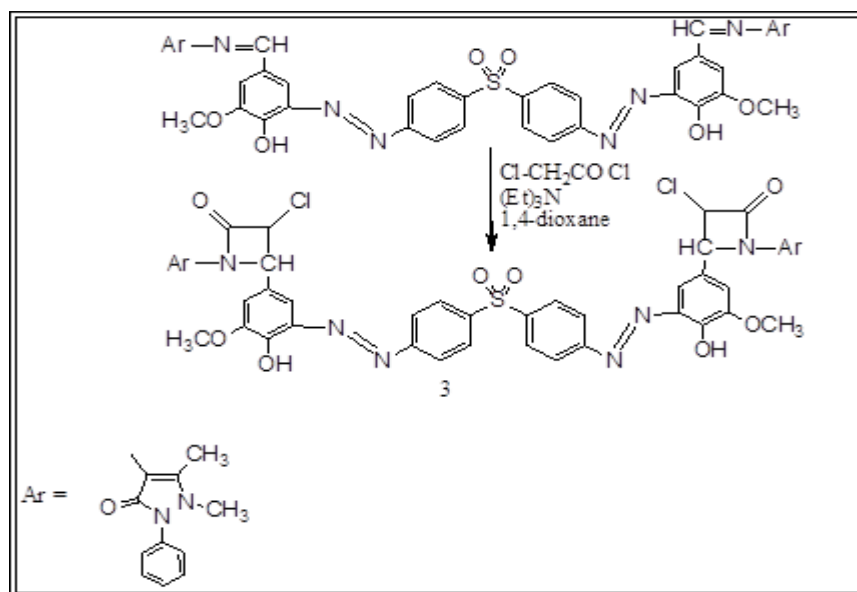
The structure of the product is important on the (FT.IR) spectroscopy of Schiff bases (2) fig(2) showed the appearance of the absorption bands in the region( $1639.49\text{cm}^{-1}$ )due to stretching vibration for azomethine group( $\text{HC}=\text{N}-$ ).With the disappearance group( $\text{CHO}$ )aldehyde at( $1691.57\text{cm}^{-1}$ ).And( $-\text{CH}$ )aromatic band at( $3064.89\text{cm}^{-1}$ ). And ( $\text{CH}$ ) aliphatic bands at( $2968.45\text{-}2935.66\text{cm}^{-1}$ ).Besides to band at( $3415.93\text{cm}^{-1}$ ) absorption ( $\text{OH}$ )phenolic

$^1\text{H}$  NMR spectrum fig(5) appeared that ( $\delta 13.6\text{ppm}$ ,s, $\text{OH}$ ) ( $\delta 9.7\text{ppm}$ ,s, $\text{CH for-N}=\text{CH}$ ), ( $\delta 7.42\text{-}8.1\text{ppm}$ ,m, $\text{CHAr}$ ), ( $\delta 7.4\text{ppm}$ ,s, $\text{CDCl}_3$ ), ( $\delta 3.9\text{ppm}$ ,s, $\text{O-CH}_3$ ), ( $\delta 3.2\text{ppm}$ ,s, $\text{N-CH}_3$ ) and ( $\delta 2.3\text{-}2.8\text{ppm}$ ,s, $\text{C-CH}_3$ ).

$^{13}\text{C}$  NMR spectrum fig(8) appeared that ( $\delta 157\text{-}161\text{ppm}$ ,Cfor- $\text{N}=\text{C}$ ), ( $\delta 101\text{-}150\text{ppm}$ , $\text{CAr}$ ) and ( $165\text{-}169\text{ppm}$ , $\text{OH}$ ); at( $76\text{ppm}$ ,solvent $\text{CDCl}_3$ ), ( $\delta 56\text{ppm}$ ,s, $\text{O-CH}_3$ ), ( $\delta 35.8\text{ppm}$ ,s, $\text{N-CH}_3$ ) and ( $\delta 10.1\text{ppm}$ ,s, $\text{C-CH}_3$ ).

**Synthesis and Characterization of compounds  $\beta$ -lactam**

These compounds were synthesized according to the sequence in Scheme (3).

Scheme (3) preparation of  $\beta$ -lactam

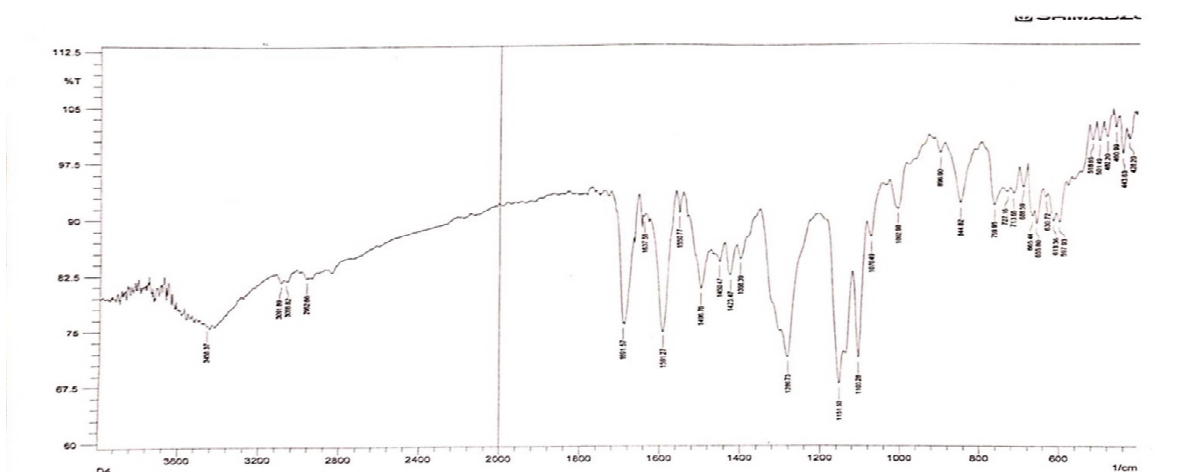
The Schiff base (2) were reaction with (chloroacetylchloride) and triethylamine. These compounds were characterized by their melting points, FT-IR,  $^1\text{H}$  NMR,  $^{13}\text{C}$ -NMR spectra and checked by TLC.

The FT-IR spectra fig(3) of compounds (3), show of carbonyl group band at  $(1786.08)\text{cm}^{-1}$ ,  $(-\text{CH})$  aromatic band at  $(3064.89)\text{cm}^{-1}$ ,  $(\text{C}-\text{Cl})\text{cm}^{-1}$  at  $(844.82)\text{cm}^{-1}$ ,  $(\text{CH})$  aliphatic bands at  $(2947.23)\text{cm}^{-1}$ , at  $(3431.36\text{ cm}^{-1})$  to OH group. and also bands at  $(1296.16)\text{cm}^{-1}$  return to the  $(\text{C}-\text{N})$  group.

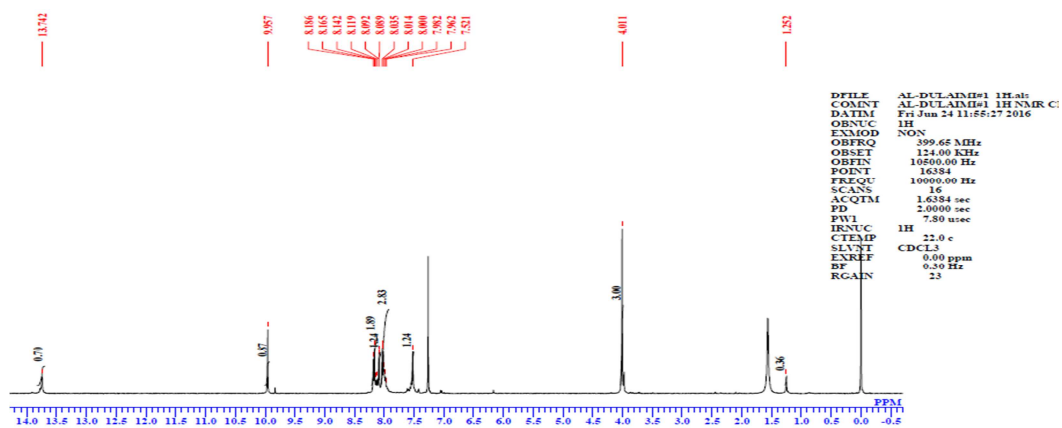
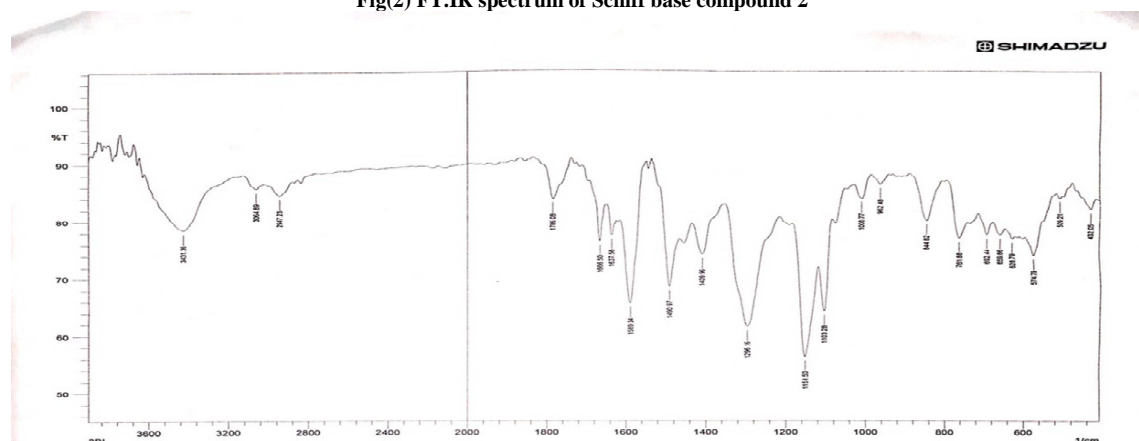
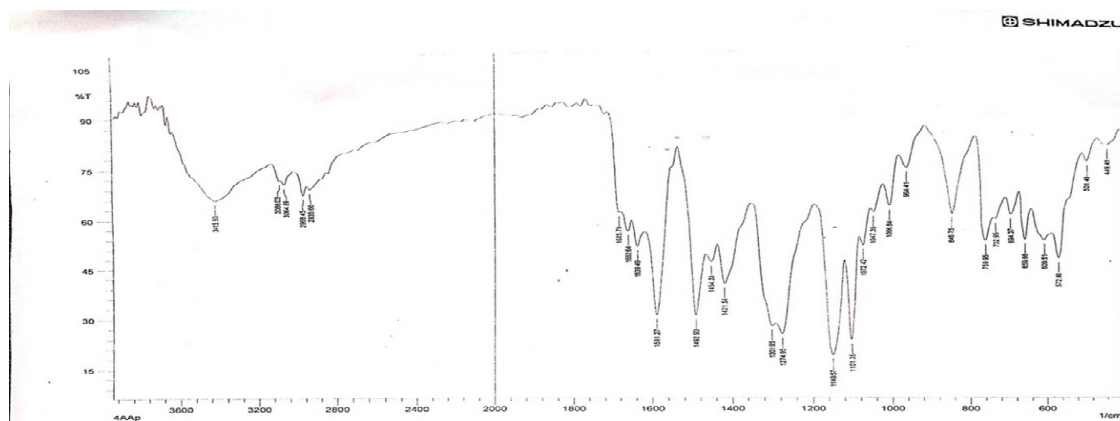
$^1\text{H}$  NMR spectrum fig(6) shows the following characteristic chemical shifts for compound (3), at  $(7.3)\text{ppm}$  to  $(\text{CDCl}_3)$ , the proton in  $(\text{HC}-\text{N})$  appeared signal at  $(9.7)\text{ppm}$ , the proton  $(\text{O}-\text{CH}_3)$  appeared at  $(3.9)\text{ppm}$ , the proton  $(\text{CH}-\text{Cl})$  appeared at  $(4.4)\text{ppm}$ , and  $(7.3-8.1)\text{ppm}$  for aromatic ring proton.

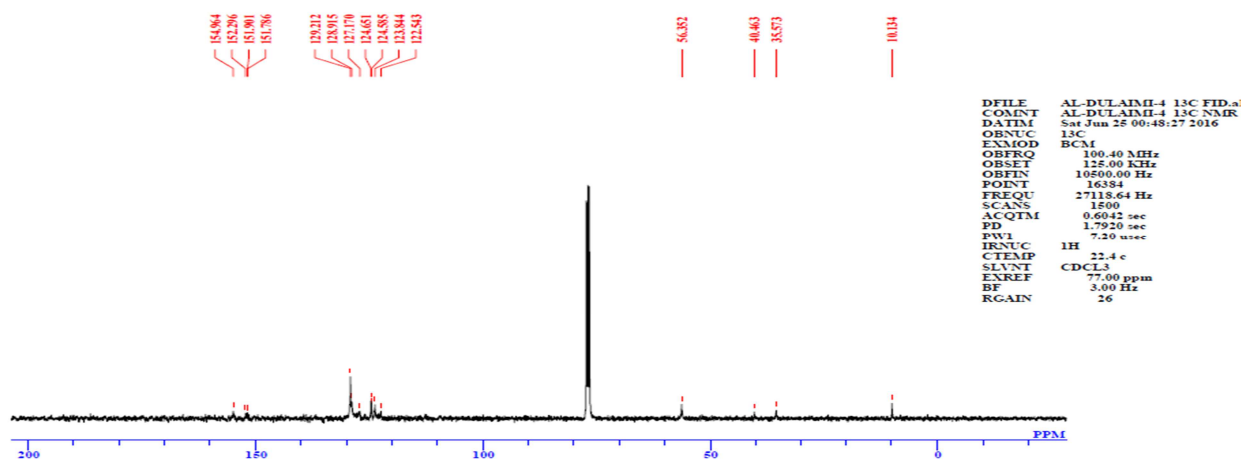
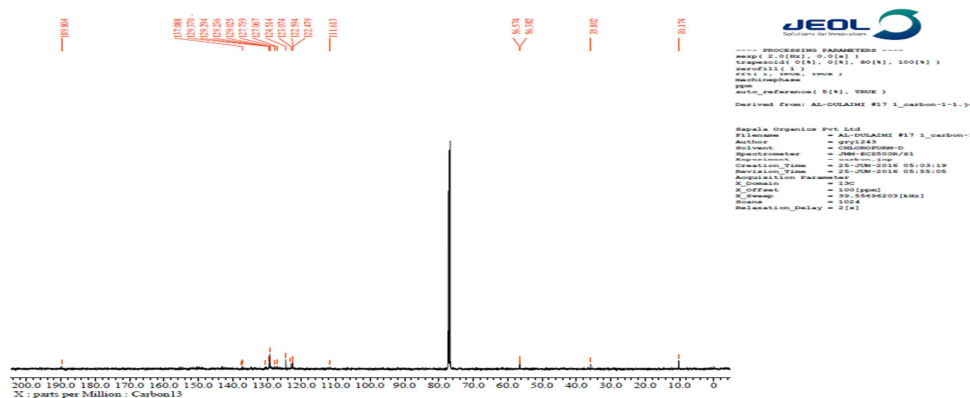
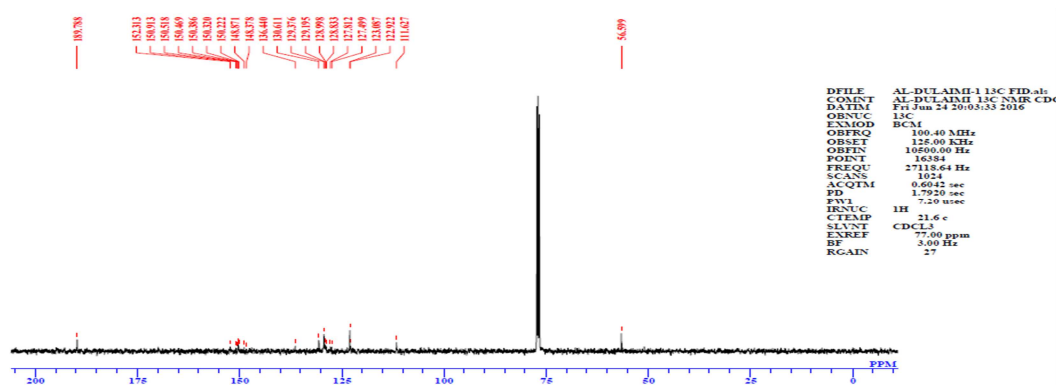
$^{13}\text{C}$ -NMR spectrum fig(9) of compound (3) showed the signal in  $(10.1)\text{ppm}$  for  $(\text{C}-\text{CH}_3)$  at  $(40.4)\text{ppm}$  due to the carbon fused with chlorine in azetidinone ring and at  $(35.5)\text{ppm}$  for the carbon of  $(\text{N}-\text{CH}_3)$  group. the carbon of  $(\text{O}-\text{CH}_3)$  at  $(56)\text{ppm}$ , the carbon of  $(\text{C}=\text{C Ar.})$  at  $(122)\text{ppm}$

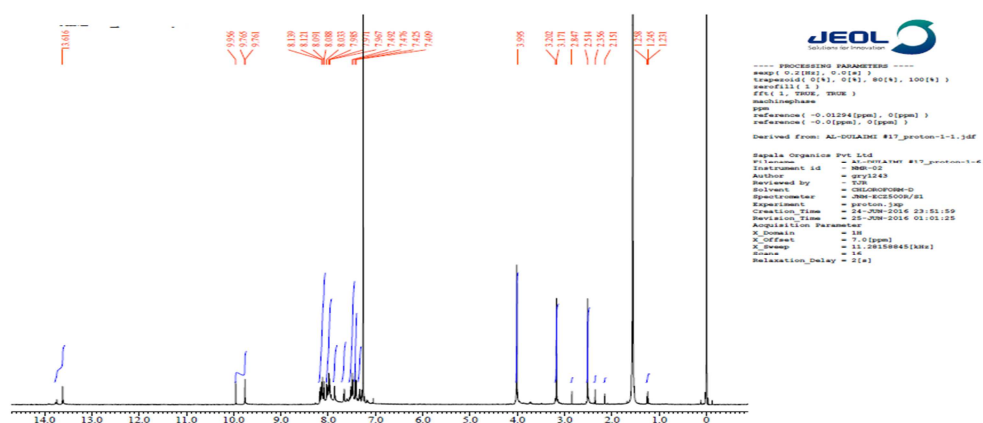
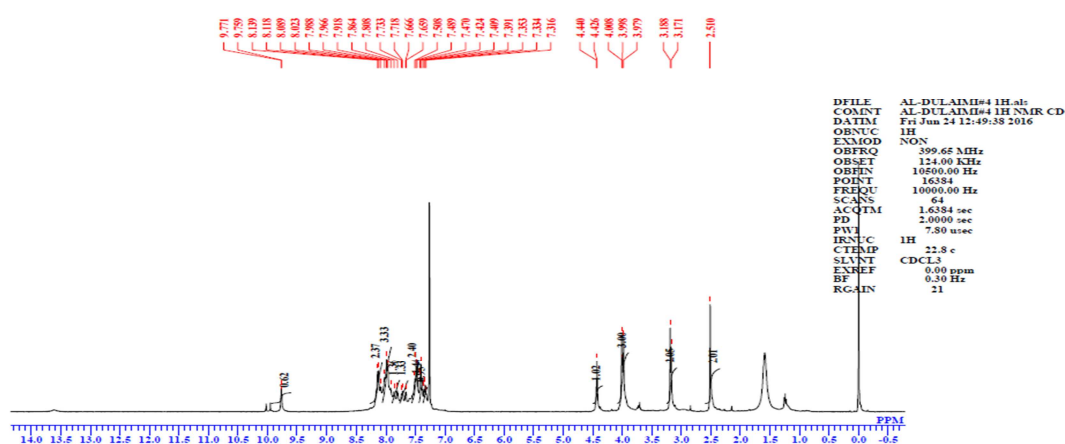
The suggested mechanism of these compounds in scheme(3).



Fig(1) FT-IR spectrum of azo compound 1





Fig(5)  $^1\text{H}$  NMR spectrum of Schiff base compound 2Fig(6)  $^1\text{H}$  NMR spectrum of  $\beta$ -lactam compound 3

## CONCLUSION

In the present study preparation of four member ring heterocyclic compounds as  $\beta$ -lactam ring, which are characterized by the spectral measurements (IR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR)

We conclude that it is possible to be  $\beta$ -lactam antibiotics effectiveness of the compounds is vital in the future compared with drugs that contain derivatives  $\beta$ -lactam.

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