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Synthesis and characterization of heterocyclic compounds from amine derivative

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Abstract : This article involves synthesis of some of heterocyclic compounds from amine derivative, the first step involves preparation of the Schiff base from benzaldehyde derivatives(2-Hydroxybenzaldehyde and 4-Bromobenzaldehyde)with 4,4-diaminodiphenyl-sulphon(DDS).These were used as precursor for the synthesis of heterocyclic compounds. Then prepared Oxazepine derivatives and Oxazepane derivatives. From Schiff base with(maliec, phthalic and succinic)anhydride.The heterocyclic compounds ware characterized by melting point,FT.IR,^{1}H NMR and C^{13}NMR.
Key Words: Heterocyclic compounds. Schiff base. 1,3-Oxazepine. 1,3-Oxazepane.

1-Introduction

The compounds containing azomethine group (-HC=N-) are known as Schiff bases, which were first reported by Hugo Schiff in 1864 and formed by condensation of a primary amine with an active carbonyl compound, and generally take place under acid, base catalysis or with heat[(1)]. Its important of ligands in coordination chemistry and find extensive application in different fields[(2,3)]. Schiff-base is associated with antibacterial[(4)], antifungal[(5)] ,antiviral[(6)], and anticancer [(7)] activities and have diverse biological activities[(8-10)].1,3-Oxazepine is unsaturated seven-membered heterocyclic containing oxygen atom at first position, nitrogen atom at third position as well asfive carbon atoms[(11)].1,3-oxazepine ring was classified as (2+5) → 7 cyclo addition reaction in which two atoms of imine group as two membered component was added to five-membered component such as (maleic or phthalic) anhydrides to give a seven membered heterocyclic[(12)].1,3-Oxazepane, Its same to the 1,3-Oxazepine but its saturated compounds and prepared by reaction imine group with succinic anhydride [(13)].

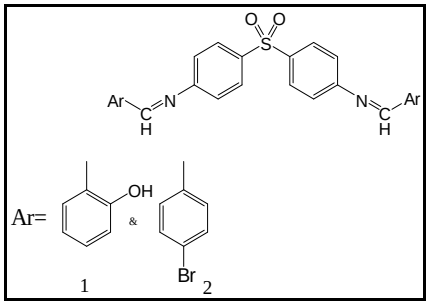
The present work includes modification of (4,4-diaminodiphenylsulphon)drug by synthesis, characterization of(4,4-diaminodiphenylsulphon)Schiff base derivative.

2-Experimental:

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}H NMR&C^{13}NMR spectra was used ($CDCl_3$).

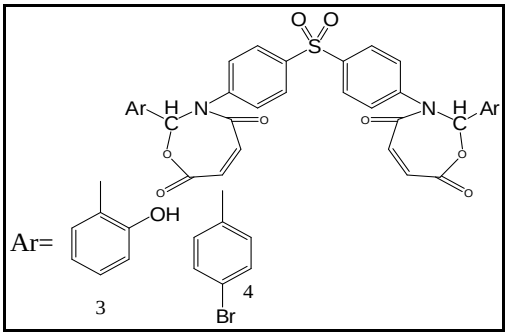
General procedure for Synthesis of Schiff bases[14] .

To a stirring solution of compound(4,4-diaminodiphenylsulphon)(0.005 mol) in absolute ethanol (20ml),the appropriate aldehyde(0.02 mol.) was added, then the mixture was refluxed, after that; cooled at room temperature. A precipitate was formed. That are filtered to afford Schiff bases compound and recrystallization of the product take place by ethanol.



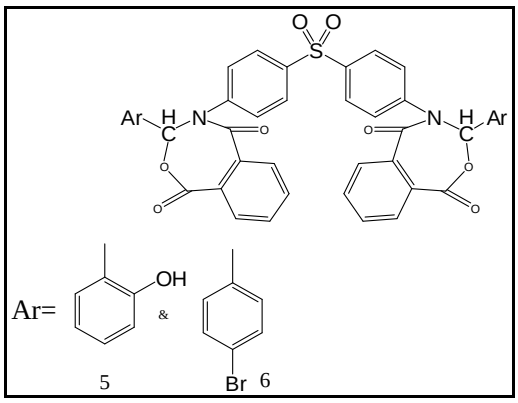
General procedure for Synthesis of oxazepine compounds maliec anhydride [15] .

A compound of schiff base(1)(1 gm) and maliec anhydride(2gm)were dissolved in dry benzene (15 ml) under reflux in oil bath(60-65)C^{o}, A precipitate was formed. That are filtered under cool condition to afford oxazepine compound. and recrystallization of the product take place by ethanol.



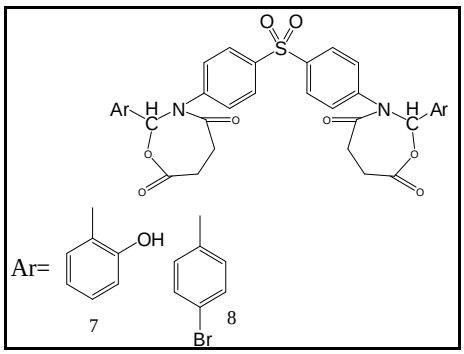
General procedure for Synthesis of oxazepine compounds from phthaliec anhydride [16].

A compound of schiff base(1-2)(1gm)andphthaliec anhydride (2gm) were dissolved in dry benzene (15 ml) under reflux for 5h in oil bath (60-65) C^{o}, A precipitate was formed. That are filtered under cool condition to afford oxazepine compound . and recrystallization of the product take place by ethanol .



General procedure for Synthesis of oxazepane compounds[17].

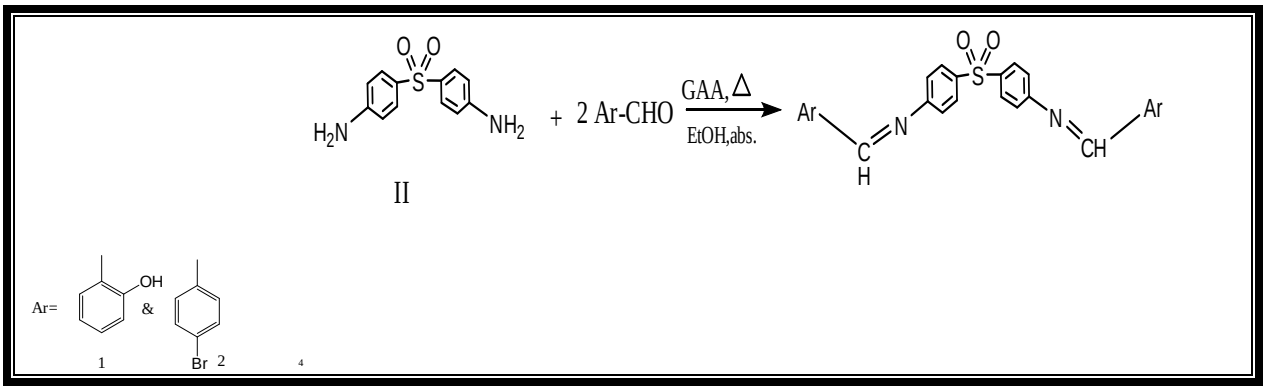
A compound of schiff base(1-2)(1 mol) and succinic anhydride (2mol) were dissolved in dry benzene (15 ml) under reflux in oil bath (60-65) C^{o}. A precipitate was formed. That are filtered under cool condition to afford oxazepane and recrystallization of the product take place by ethanol.



3-Results and discussion :-

Synthesis of Schiff base compounds(1-2)

Schiff base compounds was prepared by condensation of 4-4-diaminodiphenylsulphon(DDS)with Aldehyde derivatives(2-hydroxy-benzaldehyde,4-Bromobenzaldehyde,2-hydroxy- naphthaldehyde)in(1:2)ratio. Schiff base, which have been prepared in the following scheme**(1)**,and table**(1)**included chemical and physical properties of these compounds with melting point.



scheme (1) preparation of Schiff base (1-2)

Table(1):physical properties and other characteristics for the synthesis Schiff base derivatives (1-2)

Time	Solvent	Rf	color	M.P	M.Wt g\mol	Molecular Formula	No.
.....	**.....**	**...**	**White**	**176-179**	**248.31**	**$C_{12}H_{12}N_2O_2S$**	**II**
3hrs	Abs.EtOH	0.3	orange	268-270	456.51	$C_{26}H_{20}N_2O_4S$	1
2o min	Abs.EtOH	0.5	White	238-240	582.31	$C_{26}H_{18}Br_2N_2O_2S$	2

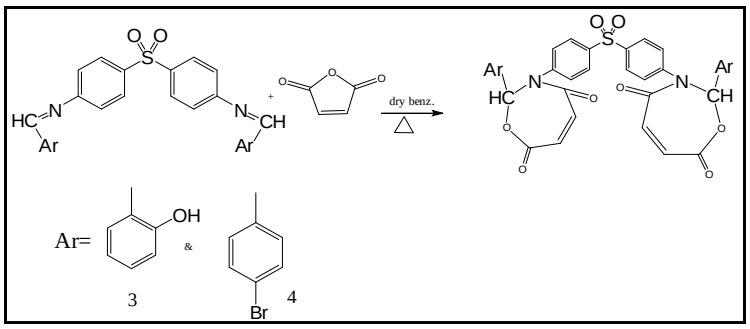
In the FT-IR spectrum there arethree major peaks; which are depending upon the different substitution groups appeared in the compound, and its relative to azomethen groups(N=CH-),(=CHAr)and(C-OH,C-Br)groups .

these compounds contain(-N=CH-)and($=CH_{Ar.}$); there are three different peaks which are appeared at(1616-1627 cm^{-1});(3051-3095cm^{-1}) respectively, and (C-OH)groups at(3228-3482cm^{-1})for compounds**(1).**appeared peak at(655cm^{-1})for(C-Br) groups .

^{1}H NMR spectrum appeared that (δ8.8-9.3ppm,s, CH for-N=CH),(δ6.6-8ppm,m,$CH_{Ar.}$)and(14.8-12.6ppm,s,OH).^{13}CNMR spectrum appeared that(δ157-161ppm,Cfor-N=C),(δ101-150ppm,$C_{Ar.}$) and(165-169ppm,OH);at(76-79ppm,solvent $CDCl_3$).

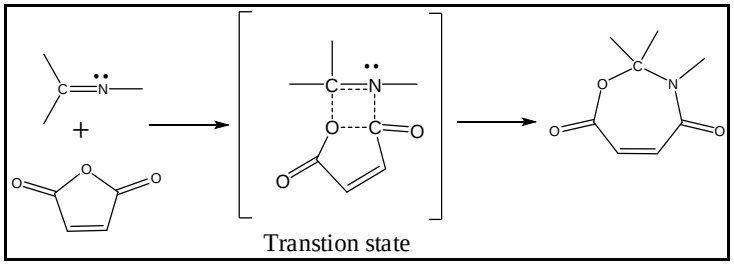
Synthesis of oxazipene compounds

Compounds**(3,4)**were synthesized from react of**(1,2)**compounds of Schiff base with maleic anhydride in dry benzene as solvent. according to the Synthetic scheme**(2)** and mechanism of oxazipene compounds



Scheme (2) preparation of oxazepine compounds

These compounds were studied and characterized by their melting points and FT-IR,^{1}HNMR, ^{13}CNMRspectra,and checked by T.L.C.



The FT-IR spectrum of these compounds shown that disappearance of (-N=CH-) group at (1616-1627cm^{-1})and its appeared new peak at(1705-1722cm^{-1}) which is relative to the lactone group(O—C=O), and its appeared new peak at 1664 cm^{-1}which is relative to (N—C=O) group.($CH_{Ar.}$) appeared at (3051-3059cm^{-1}) and OH group appeared at (3213-3520cm^{-1}).

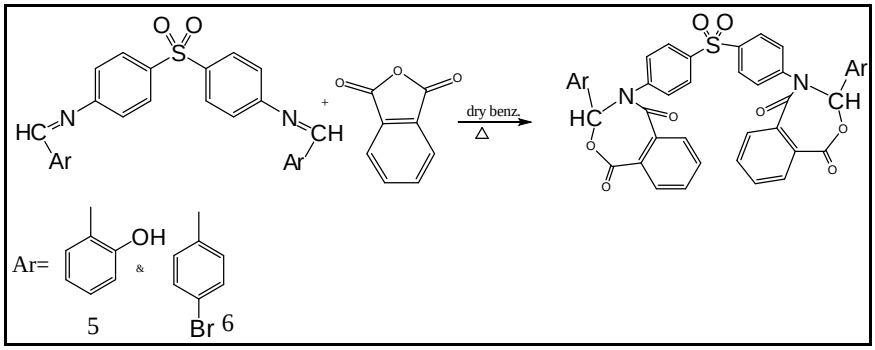
^{1}H NMR spectrum chart appeared that :(δ3.3ppm,solvent H_2O,D_2O), (δ2.5ppm,solvent DMSO),at(δ9.6-8.6ppm,s,CH_{cycle}),at(δ6.9-8.1ppm,m,$CH_{Ar.}$),at(6.3-6.97ppm,d,$CH_{olfenic}$);and(δ12.6-15,2 ppm,s,OH).

^{13}CNMR spectrum appeared that(δ160-162ppm,C forN—C), (δ152-155ppm,C for C=C_{cycle});(δ160-163ppm,C forC—O);(δ190-191ppm,C for C=O Lactam);(δ160-162ppm,C forC=O Lactone);(δ112-138ppm,

$C_{Ar.}$)and(165ppm,C for C—OH);at(δ 39ppm,solvent $CDCl_3$).

Synthesis of oxazepine compounds

Compounds**(5,6)**were synthesized from react of**(1,2)**compounds of Schiff base with phthalic anhydride in dry benzene as solvent . According to the Synthetic scheme**(3)**.



scheme (3) preparation of oxazepine compounds

These compounds were studied and characterized by their melting points and FT-IR,^{1}H NMR,^{13}CNMR spectra, and checked by T.L.C.

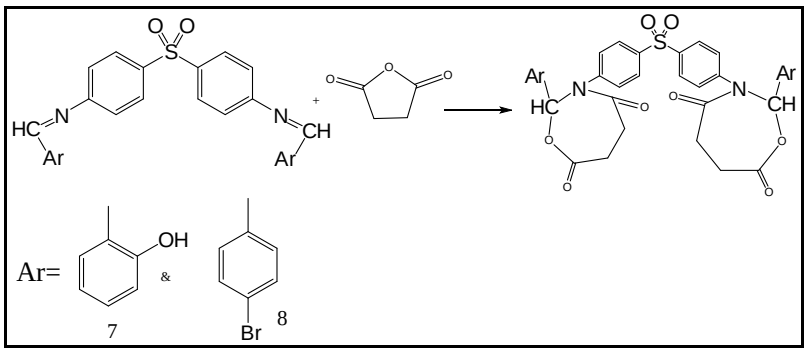
The FT-IR spectrum of these compound shown that disappearance of (-N=CH-) group at (1616-1627cm^{-1}) and its appeared new peak at (1687-1722cm^{-1}) which is relative to the lactone group (O—C=O), and its appeared new peak at (1637-1695cm^{-1}) which is relative to (N—C=O) group .($CH_{Ar.}$) appeared at(3062-3091cm^{-1})and OH group appeared at(3250-3550cm^{-1}) for compounds**(5,6).**

^{1}H NMR spectrum chart appeared that : (δ3.3ppm,solvent H_2O,D_2O), (δ2.5ppm,solvent DMSO),at(δ8.3-9.9ppm,s,CH_{cycle}),at(δ6.9-8.5ppm,m,$CH_{Ar.}$);and(δ12.6-15.2 ppm,s,OH).

^{13}CNMR spectrum appeared that(δ155ppm,C forN—C);(δ160-155ppm,C forC—O);(δ160ppm,C for C=O Lactam);(δ190ppm,C forC=O Lactone);(δ114-138ppm, $C_{Ar.}$)and at(δppm, solvent $CDCl_3$).

Synthesis of oxazipane compounds

Compounds**(7,8)** were synthesized from react of **(1,2)** compound of Schiff bases with succinic anhydride in dry benzene as solvent. According to the Synthetic scheme(4).



Scheme (4) preparation of oxazepane compounds

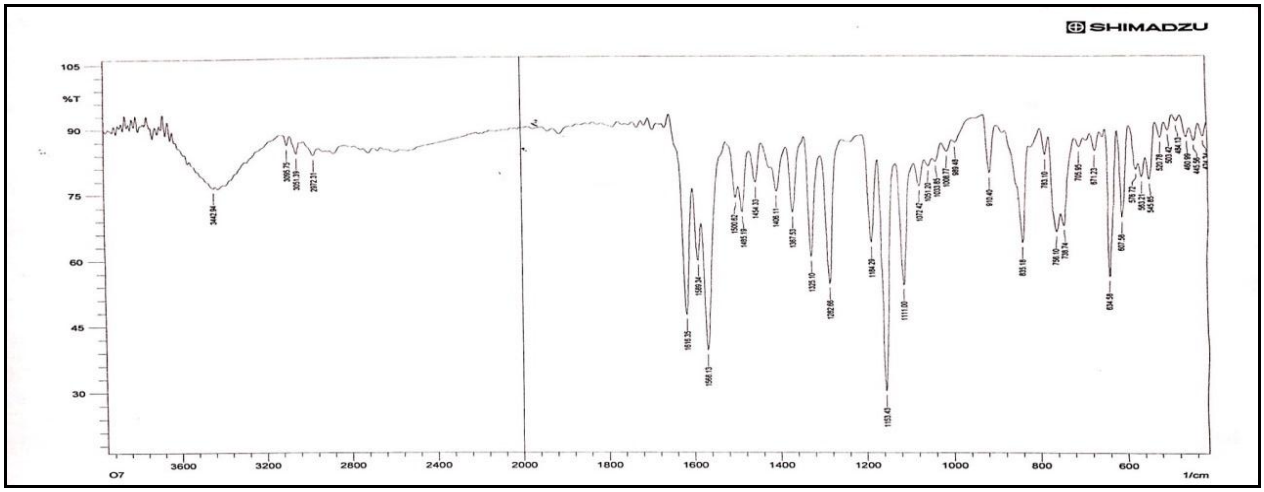
These compounds were studied and characterized by their melting points and FT-IR, ^{1}H NMR, ^{13}CNMR spectra, and checked by T.L.C.

The FT-IR spectrum of these compound shown that disappearance of (-N=CH-) group at (1616-1627cm^{-1}) and its appeared new peak at (1695-1714cm^{-1}) which is relative to the lactone group (O—C=O), and its appeared new peak at (1666-1672cm^{-1}) which is relative to (N—C=O) group .($CH_{Ar.}$) appeared at(3051-3091cm^{-1})and OH group at(3280-3510cm^{-1}) for compounds**(7).**

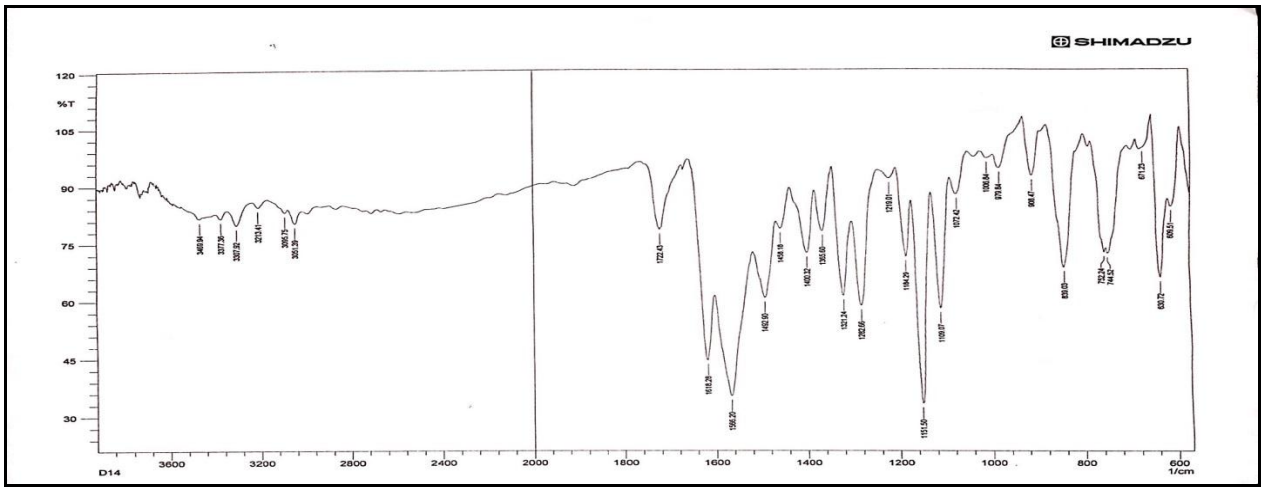
^{1}H NMR spectrum chart appeared that : (δ3.3ppm,solvent H_2O,D_2O),(δ2.5ppm,solvent DMSO),at(δ8.6-9.3ppm,s,CH_{cycle}), at (δ7-8.1ppm,m,$CH_{Ar.}$), at(6.3-6.97ppm,d,$CH_{olfenic}$) at (δ1.2ppm,t,CH_2CON), at (δ1.7ppm,t,CH_2COO); and(δ12.6-14.8ppm,s,OH).^{13}CNMR spectrum appeared that(δ143ppm,C forN—C);(δ21-28ppm,C forC—O);(δ171ppm,C for C=O Lactam);(δ173ppm,C forC=O Lactone); (δ118-137ppm, $C_{Ar.}$)and at(δppm,solvent $CDCl_3$).

Table (2) : physical properties and other characteristics for the synthesis1,3-Oxazepine&1,3-Oxazepane(3-8)

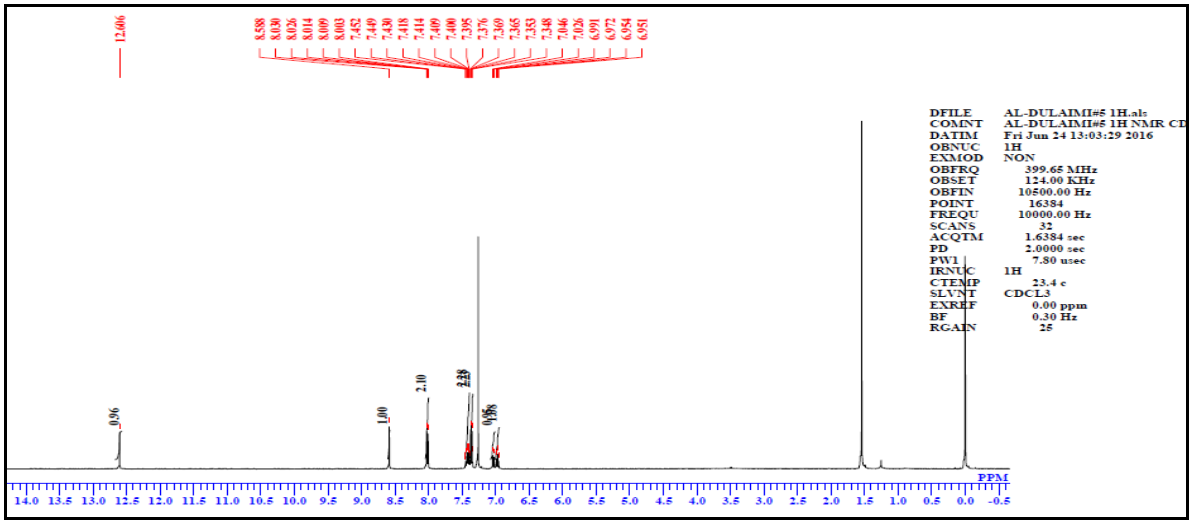
Time	Solvent	Rf	color	M.P	M.Wt g\mol	Molecular Formula	No.
9 hrs	toluene	0.54	Yellow	253-255	652.63	$C_{34}H_{24}N_2O_{10}S$	3
9:30hrs	Benzen	0.56	White	203-205	778.42	$C_{34}H_{22}Br_2N_2O_8S$	4
11:30 hrs	toluene	0.35	Yellow	269-272	752.74	$C_{42}H_{28}N_2O_{10}S$	5
9:30hrs	Benzen	0.72	White	235-239	878.54	$C_{42}H_{26}Br_2N_2O_8S$	6
10hrs	benzen	0.4	yallow	265-267	656.66	$C_{34}H_{28}N_2O_{10}S$	7
10 hrs	benzen	0.6	white	218-221	782.45	$C_{34}H_{26}Br_2N_2O_8S$	8



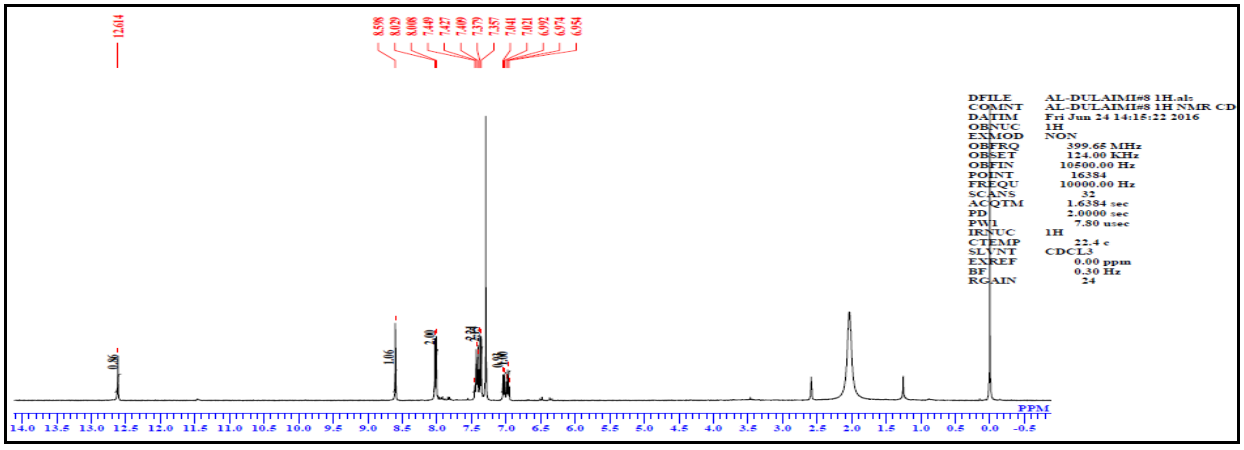
Figure(1):FT.IR spectra for compound1



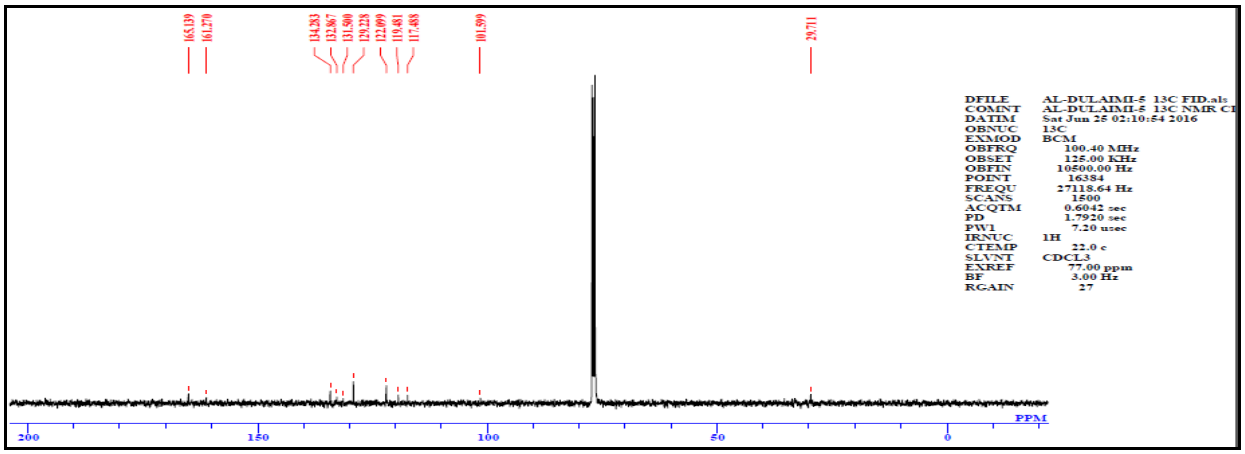
Figure(2):FT.IR spectra for compound3



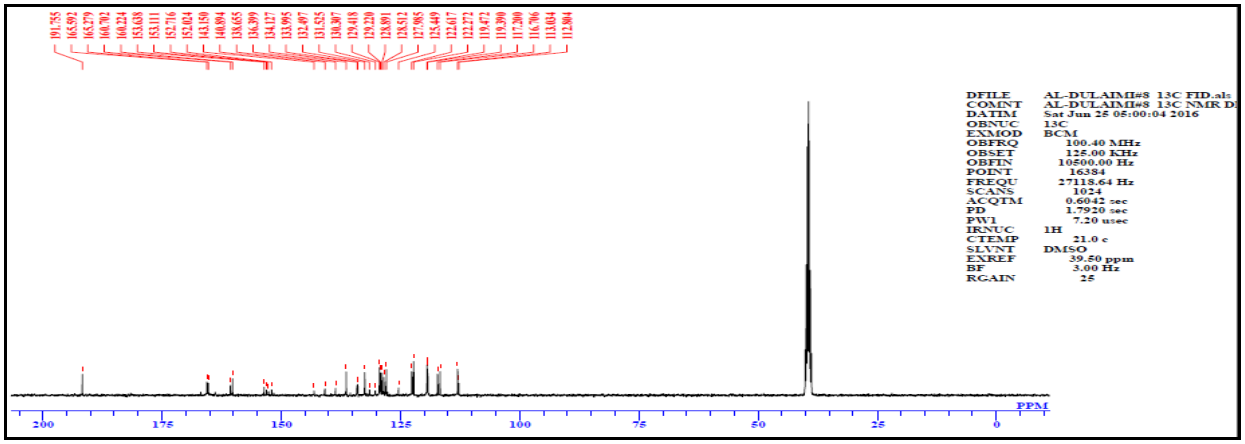
Figure(3):^{1}H NMR spectra for compound 1



Figure(4):^{1}H NMR spectra for compound 3



Figure(5):^{13}C NMR spectra for compound 1



Figure(6):^{13}C NMR spectra for compound 3

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