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Synthesis And Characterization Of Novel 1,3-Oxazepin-4-Ones Derivatives Via Schiff Bases Reactions With Phthalide

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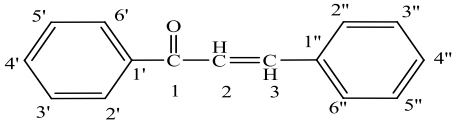
ABSTRACT

A number of novel 1,3-oxazepin-4-ones derivatives were synthesized by acid-catalyzed cycloaddition reaction of Schiff bases with Phthalide in anhydrous THF under dry and reflux conditions with high yields. Schiff bases were synthesized by thermal condensation reaction of aromatic aldehydes, ketones or prepared chalcones with aromatic primary amines. The products were identified by their melting point, FT-IR, UV-Vis., ^{1}H-NMR and ^{13}C-NMR spectra.

Introduction

Chalcones are class of naturally occurring and synthesized compounds of great importance due to their uses as precursors and key intermediates for organic and bio-organic, heterocyclic systems and organometallic synthesis and other applications such as optical materials, UV-absorbing filters, holographic papers, liquid crystal components and food industry.[1-4] The genral structure is represented by figure (1).



Figure(1) Chalcone structure.

Chalcones possess a wide spectrum of biological activities and pharmacological applications such as anti-cancer, anti-bacterial, anti-microbial, anti-tubercular, anti-viral, anti-malaria, anti-leis mania, anti-ulcerative anti-oxidant, anti-hyperglycemic, anti-inflammatory, analgesic anti-diabetic,(5-8) in addition to their uses as insecticides and pesticides.[9]

Chalcones have been synthesized by different methods, of these are Cleisen-Schmidt, Friedel-Craft acylation, Suzuki coupling reaction Wittig reaction, and Von-Konstanecki and Ganguly methods. [10-13]

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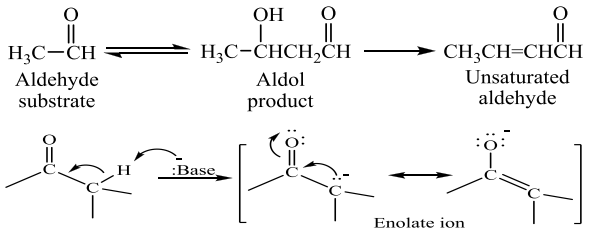
The base-catalyzed Cleisen-Schmidt reaction is involving carbonyl condensation reaction of methyl ketones with aldehydes (aldol condensation) to produce the enolate ion in equilibrium with the carbonyl compound which reacts further to form an aldol product associated with expulsion of a small molecule such as water or alcohol to form the α,β-unsaturated aldehydes or ketones.[14]



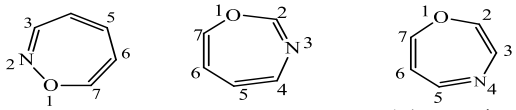
Schiff bases (imines) are class of compounds containing the azomethine group (-HC=N-) , usually prepared by the condensation of amino group in primary amines, amino acids and hydrazines with an active carbonyl of aldehydes and ketones. The first classical synthetic route was reported by Hugo Schiff in 1864 that involves thermal condensation of amino group with carbonyl group associated with simultaneous removal of water via a zeotropic distillation.[15]

Schiff bases are versatile precursors in the synthesis of organic, bio-organic, organometallic, heterocyclic, protective agents and industrial compounds via ring closure, cycloaddition and replacement reactions. In addition Schiff bases are used in analytical, material, catalysis and magneto chemistry, photo physical studies and oxygen

transporter. [16-17] They exhibit a wide range of biological activities and pharmacological applications such as anti-microbial[18], anti-parasitic[19] anti-inflammatory[20], anti-cancer anti-tumor, anti-fungal ,ant-leukemia activities. [21-25]

Phthalides are class of secondary metabolites or phytochemical compounds classified as lactones,[26] which are known to be stimulating and/or inhibiting agents for various enzymes in the body, and help to lower blood pressure . [27] Many of the phthalides have been isolated[28], and used as versatile starting materials for the synthesis of variety of organic and bio-organic, carbo- and heterocyclic compounds and key intermediates for the synthesis of natural products.[29] Some 3- alkyl / aryl substituted phthalides have been used as starting materials for the synthesis Phthalides have been conveniently prepared by refluxing o-aroyl benzoic acid with substituted / unsubstituted bromo derivatives of acetophenone, propiophenone, coumarinyl acetophenone or diethyl bromomalonate in ethyl methyl ketone in the presence of K_2CO_3. Their anti-microbial, analgesic, anti-bacterial, anti-fungal and anti-oxidant behaviors were extensively screened. [30]

Oxazepines are class of heterocyclic compounds of seven- membered ring with two hetero- atoms (O&N), oxygen atom is located at position (1) and nitrogen atom in the (-2,-3 or-4) positions.[31]



Figure(2):Structures of oxazepines

Oxazepines have been synthesized mainly by dipolar cycloaddition reaction of imines with five atoms cyclic anhydride, such as maleic, succinic, phthalic and from photochemical ring expansion reactions of pyrimidines and aziridines.[32-35] They possess a wide range of biological activities and

pharmacological applications such as anti-depressant, analgesic, psychoactive drug, anti-tumor anti-convulsant, enzyme inhibitor, anti-histaminic, anti-allergic, anti-bacterial, anti-microbial, anti-oxidant anti-fungal and anti-inflammatory, beside their uses as corrosion inhibitors, polymers photo stabilizers and liquid crystal components. [36-41]

Experimental Part

Melting points were recorded on Electro thermal Melting Point Apparatus (uncorrected).FT-IR spectra were recorded at room temperature from (4000-400) cm^{-1} with KBr disc on Infrared Spectrophotometer Model Tensor 27 Bruker Co., Germany, and UV-Vis. spectra were recorded at R.T from(200- -400) nm in absolute ethanol on Shimadzu Double-Beam Spectrophotometer UV-210 A. The ^{1}H-NMR and ^{13}C-NMR spectra were recorded on Bruker Ac-300MHz spectrometer.

Syntheses of chalcones (H_1, H_2& H_3).

Chalcones were synthesized according to literature procedur. [14] A mixture of 100 ml pure benzaldehyde and 38ml of acetone was cooled to approximately 10°C. This mixture was gradually added to 40ml (40%) cold ethanol-NaOH solution in two separate portions, (approximately 5 minutes apart) with continuous stirring for 30 minutes. Then the mixture was poured onto the ice-water mixture whereupon a crystalline product was formed which was filtered off ,washed with distilled water and recrystallized from water-ethanol mixture and dried.

Synthesis of Schiff bases (M_1-M_9).

Schiff bases were synthesized according to literature procedur.[33] An equimolar mixtures (0.02mol), of aldehydes and aromatic amines and trace of glacial acetic acid as catalyst in absolute ethanol (25ml) was placed in a (100ml) round-bottom flask equipped with condenser and stirring bar. The mixture was allowed to react at reflux temperature for 4hr, then to cool down to room temperature, whereby a crystalline solid separated out. The solid product was filtered off and recrystallized form ethanol. The structural formuli, names, melting points, colors, and percentage yields for the synthesized Schiff bases are given in table1.

Synthesis of 1,3-oxazepin-4-ones derivatives(N_1-N_9)

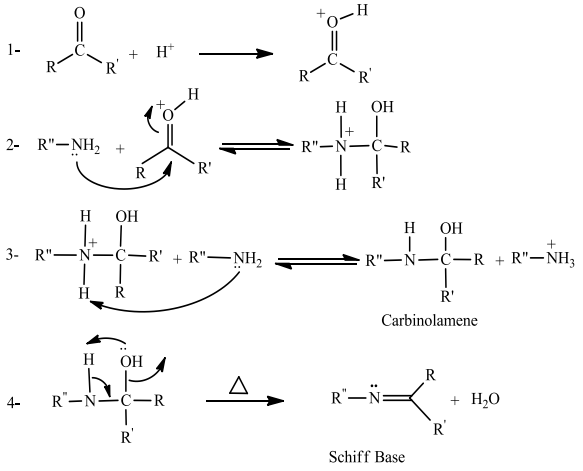
In well dried 100-ml round-bottom flask equipped with condenser and anhydrous calcium chloride tube guard a mixture of Schiff bases (0.01mol) and isobenzofuran-1(3*H*)-one (0.01mol) dissolved in (20ml) of tetrahydrofuran (THF) with trace of glacial acetic acid as catalyst was refluxed for 3hr and left to stand for 24hr at room temperature Then solid product separated out. The solid product was filtered off and recrystallized form ethanol. The structural formuli, names, melting points, colors, and

percentage yields for the synthesized 1,3-oxazepin-4-ones derivatives are given in table2.

Results and Discussion

Chalcones were synthesized from commercially available aldehydes, acetone and acetophenone and identified by their melting points, FT-IR and UV-Vis. spectra, tables, (1), (4) and (7). Formation of the products were followed up by the appearance of the stretching absorption bands in the FT-IR spectra of; (C=O) at (1652-1668) cm^{-1}, (C-H arom.) at (3052-3082) cm^{-1} beside the characteristic stretching absorption bands of the residual groups in the structure, table, (7). The UV-Vis. spectra of these chalcones showed absorption maxima at (228-350) nm owing to the electronic transfers π-π* and n-π* characteristic of the structures of the synthesized chalcones (H_1-H_3).

Schiff bases were synthesized from commercially available aldehydes, ketones and synthesized chalcones with primary amines and identified by their melting points, FT-IR and UV-Vis. spectra, tables, (2), (5) and (8). The FT-IR spectra showed the appearance of the stretching absorption bands of the characteristic groups of the resulting imines: azomethine (C=N) at (1584-1651) cm^{-1} beside the characteristic bands of the residual groups in the structure, table, (8) indicative of formation of the products. The UV-Vis. Spectra of these imines showed absorption maxima at (205-312) nm owing to the electronic transitions: π-π* and n-π* characteristic of the structures of the synthesized imines (M_1-M_9). The mechanism of Schiff bases formation, scheme(1), was thoroughly studied and established by many authorized literatures[42-44].



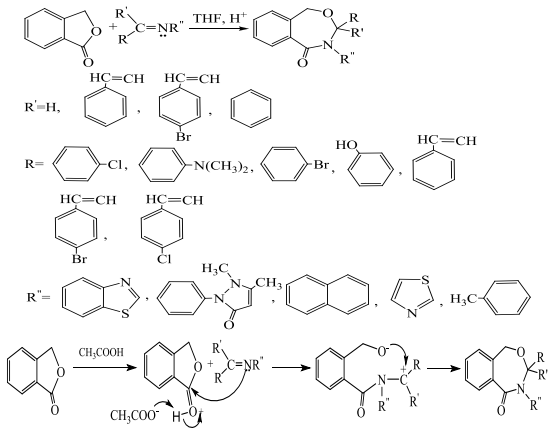
Scheme (1): Mechanism of Schiff bases formation.

In this work the synthesis of novel 1,3-oxazepin-4-ones derivatives by direct reaction of several Schiff bases with Phthalide in dry THF is reported. The synthesis of these compounds were achieved by the reaction of imines and Phthalide in anhydrous THF at dry and reflux conditions. The resulting products were identified by their melting points, FT-IR and UV-Vis. spectra, tables, (3), (6) and (9). The FT-IR spectra of the products showed characteristic stretching absorption bands at (1649-1701) cm^{-1} indicative of C=O (lactone) bond formation beside the characteristic stretching absorption bands of the residual groups in the structure, Figures (3) and (4). The UV-Vis. spectra showed absorption maxima at (235-364) nm owing to the electronic transitions: π-π* and n-π* characteristic of the structure of the synthesized 1,3-oxazepin-4-ones derivatives.

The ^{1}H-NMR spectrum of compound N_1 in solvent $CDCl_3$ showed chemical shifts, δ(ppm): singlet 5.30 (2H, O-$\underline{CH_2}$), singlet 10.01 (H, N-$\underline{CH}$), multiplet 7.06-7.98 (12H, aromatic protons), and spectrum of compound N_2 showed chemical shifts δ(ppm) at: singlet 2.63 (3H, $\underline{CH_3}$), singlet 5.31 (2H,O-$\underline{CH_2}$), singlet 9.96 (1H,N-$\underline{CH}$), multplet 6.97-7.83 (12H,aromatic protons), as given in table (10) and Figures (5) and (6) of N_1 and N_2 . The ^{13}C-NMR spectrum of compound N_5 in solvent $CDCl_3$ showed chemical shifts δ(ppm) at: 21.11 (3H, $\underline{CH_3}$) 46.48 (2H, O-$\underline{CH_2}$), 69.69 (1H, N-$\underline{CH}$) 161.76 (0H, N-$\underline{CO}$), 117.25-145.94 (12H, Arom. Carbons), other chemical shifts, δ(ppm) in table (11), Figure (7) showed chemical shifts of N_5.

It may be concluded that the reaction takes place via concerted (5+2) dipolar cycloaddition mechanism in which the mild nucleophile(imine) attacked the electrophilic carbon atom of the carbonyl group to give a dipolar intermediate, which collapses to give the target molecule, the roll of the acid-catalyst is to enhance the electro positivity of the carbon nucleus.

The reaction course and the suggested mechanism is given by scheme (2).



Scheme (2): Reaction course and suggested mechanisms for the formation of 1,2-disubstituted-1,3-oxazepine-4-ones.

Table1. Structural formulas, names, melting points, colors, and % yields of chalcones. (H_1-H_3).

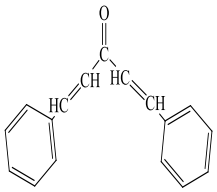
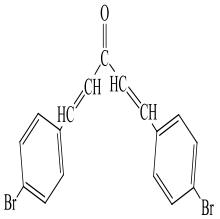
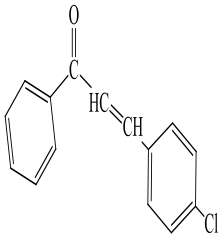
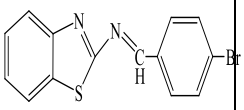
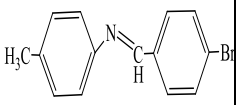
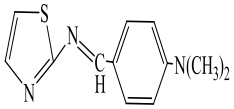
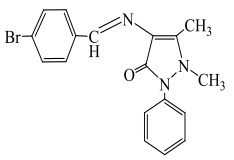
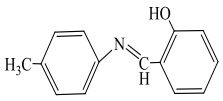
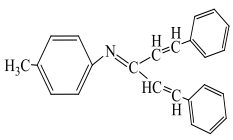
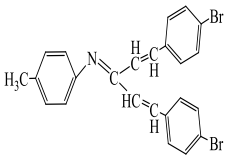
Comp. No.	Structural formula	Name	Yield %	m.p. °C	Color
H_1		1,5-diphenyl penta -1,4-dien-3-one	85%	92-95	Yellow
H_2		1,5-bis(4-bromo phenyl)penta-1,4-dien-3-one	65%	192-195	Yellow
H_3		3-(4-chloro phenyl)-1-phenylprop-2-en-1-one	87%	112-115	Yellow

Table2. Structural formulas, names, melting points, colors, and % yields of Schiff bases (M_1-M_9).

Comp. No.	Structural formula	Name	Yield %	m.p. °C	Color
M_1		*N*-(4-bromobenzylidene)benzo[*d*]thiazol-2-amine	88%	111-113	Bright Pale yellow
M_2		*N*-(4-bromobenzylidene)-4-methylaniline	92%	140-142	Bright White
M_3		*N*-(4-(dimethylamino)benzylidene)thiazol-2-amine	66%	143-145	Bright Brown
M_4		4-(4-bromobenzylideneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one	85%	253-254	Bright Pale yellow
M_5		2-((*p*-tolylimino) methyl)phenol	87%	95-96	Bright Yellow
M_6		*N*-(1,5-diphenyl penta-1,4-dien-3-ylidene)-4-methylaniline	82%	95-97	Bright Pale nutty
M_7		*N*-(1,5-bis(4-bromoph enyl)penta-1,4-dien-3-ylidene)-4-methyl aniline	72%	205-207	Bright Light tan

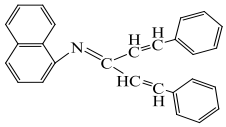
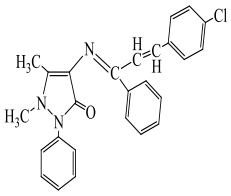
M8		***N*-(1,5-diphenyl pent a-1,4-dien-3-ylidene) naphthalen-1-amine**	**71%**	**82-83**	**Brown**
M9		**4-(1,5-bis(4-chloroph enyl)-1-phenylallylid eneamino)-1,5-dime thyl-2-phenyl-1*H*-py razol-3(2*H*)-one**	**71%**	**105-107**	**Pale orange**

Table3. Structural formulas, names, melting points, colours, and % yields of 1,3-oxazepin-4-ones derivatives (N_1-N_9).

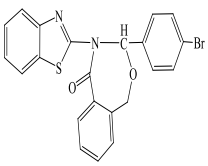
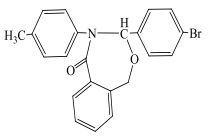
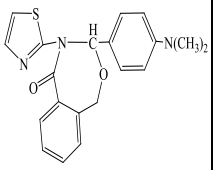
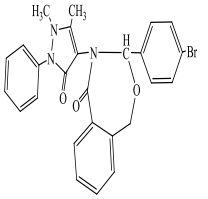
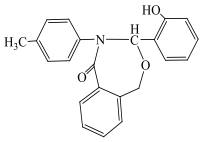
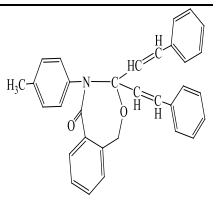
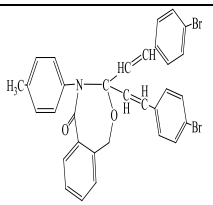
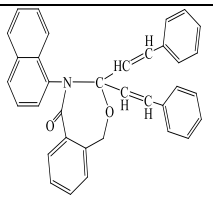
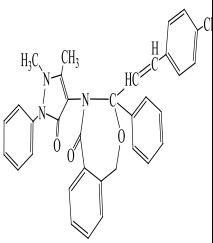
Comp. No.	Structural formula	Name	Yield %	m.p. °C	Color
N1		**4-(benzo[*d*]thiazol-2-yl)-3-(4-bromophenyl)-3,4-dihydrobenzo[*e*][1 ,3] oxazepin-5(1*H*)-one**	**91%**	**107-109**	**Bright pale yellow**
N2		**3-(4-bromophenyl)-4-*p*-t olyl-3,4-dihydrobenzo[*e*] [1,3]oxazepin-5(1*H*)-one**	**89%**	**105-108**	**Bright pale yellow**
N3		**3-(4-(dimethylamino)ph enyl)-4-(thiazol-2-yl)-3,4-dihydrobenzo[*e*][1,3] oxazepin-5(1*H*)-one**	**83%**	**63-65**	**Bright nutty**
N4		**3-(4-bromophenyl)-4-(1,5 -dimethyl-3-oxo-2-phenyl -2,3-dihydro-1*H*-pyrazol- 4-yl)-3,4-dihydrobenzo[*e*] [1,3]oxazepin-5(1*H*)-one**	**93%**	**210-212**	**Bright pale yellow**
N5		**3-(2-hydroxyphenyl)-4-*p*-tolyl-3,4-dihydrobenzo[*e*] [1,3]oxazepin-5(1*H*)-one**	**81%**	**68-70**	**Bright yellow**
N6		**3,3-distyryl-4-*p*-tolyl-3, 4-dihydrobenzo[*e*][1,3] oxazepin-5(1*H*)-one**	**87%**	**86-88**	**Pale green**
N7		**3,3-bis(4-bromostyryl)-4-*p*-tolyl-3,4-dihydrobenzo [*e*][1,3]oxazepin-5(1*H*)-one**	**82%**	**168-170**	**Bright pale yellow**
N8		**4-(naphthalen-1-yl)-3,3-distyryl-3,4-dihydrobe nzo[*e*][1,3]oxazepin-5 (1*H*) –one**	**84%**	**> 300**	**Bright pale yellow**
N9		**3-(4-chlorostyryl)-4-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*- pyrazol-4-yl)-3-phenyl-3,4-dihydr obenzo[*e*] [1,3]oxazepin-5 (1*H*)-one**	**89%**	**78-80**	**Bright pale yellow**

Table 4: The UV-Visible Absorption Max (nm) in DMSO of Chalcones (H_1-H_3).

Comp. code	Wave Length: λ/nm	
H_1	**234.0**	**350.0**
H_2	**228.0**	**344.0**
H_3	**263.0**	**312.0**

Table 5: The UV-Visible Absorption Max (nm) in Ethanol of Shiffs Bases (M_1-M_9)

Comp. code	Wave Length: λ/nm	
M_1	**208.0**	**265.0**
M_2	**215.0**	**277.0**
M_3	**220.0**	**260.0**
M_4	**222.0**	**300.0**
M_5	**209.0**	**284.0**
M_6	**215.0**	**298.0**
M_7	**218.0**	**282.0**
M_8	**230.0**	**270.0**
M_9	**205.0**	**268.0**

Table 6: The UV-Visible Absorption Max (nm) in Ethanol chloroform of 1,3-oxazepin-4-ones derivatives (N_1-N_9).

Comp. Code	Wave Length: λ/nm	
N_1	245.0	348.0
N_2	242.0	364.0
N_3	262.0	344.0
N_4	235.0	355.0
N_5	243.0	345.0
N_6	246.0	330.0
N_7	260.0	365.0
N_8	262.0	348.0
N_9	235.0	334.0

Table7. FTIR of Chalcones (H_1-H_3).

Comp. No	FT-IR(KBr), ν(cm^{-1})					
	C=O Alke.	C=C Aro.	C=C Aro.	C-H Aro.	C-H Alke.	Others
H_1	1652	1595	1573	3082	3100	--
H_2	1656	1600	1581	3052	3095	C-Br 536
H_3	1668	1576	1569	3075	3086	C-Cl 762

Table8. FTIR of Schiff bases (M_1-M_9).

Comp. No	FT-IR(KBr), ν(cm^{-1})									
	C=O	C=CAro.	C=N	C-H Eli.	C-H Aro.	C-N	=C-H Non Aro.	C=CAlke	C-Cl	Others
M_1	--	1573	1651	--	3087	1123	3094	--	--	C-S 1200 C-Br 598
M_2	--	1498	1616	2934	3095	1167	3115	--	--	--
M_3	--	1467	1634	2899	3063	1181	3080	--	--	C-S 1214
M_4	1657	1489	1627	2923	3043	1131	3066	1545	--	C-Br 575
M_5	--	1476	1651	2895	3043	1175	3064	--	--	O-H 3326b
M_6	--	1509	1584	2966	3039	1139	3056	1564	--	--
M_7	--	1512	1645	2967	3074	1167	3091	1564	--	C-Br 579
M_8	--	1463	1592	--	3083	1157	3098	1530	--	--
M_9	1699	1492	1622	2899	3104	1166	3110	1568	734	N-N 1134

Table9. FT-IR of 1,3-oxazepin-4-ones derivatives (N_1-N_9)

Comp. No	FT-IR(KBr), ν(cm^{-1})								
	C-O	C=CAro.	C-N Lactam	C=N	C-H Aro.	C-H Ali.	C-Br	N-C=O Lactam	Others
N_1	1163	1636	1205	1588	3059	2937	575	1691	C-S 1248
N_2	1187	1561	1216	--	3085	2979	587	1649	--
N_3	1181	1543	1182	1500	3108	2967	--	1700	C-S 1186
N_4	1192	1490	1191	--	3082	2965	584	1679	N-N 1148 C=C alke1590
N_5	1158	1597	1182	--	3052	2921	--	1664	-OH 3426b
N_6	1194	1572	1186	--	3082	2933	--	1693	C=C alke1604 C-H alke 3100

N_7	1162	1631	1192	--	3096	2914	562	1678	C=C alke1654 C-H alke 3108
N_8	1192	1496	1175	--	3082	2889	--	1682	C=C alke1598 C-H alke3098
N_9	1186	1582	1174		3100	2976		1701	C=C alke1620 C-H alke 3116 N-N 1152 C-Cl 742

Table 10: The ^{1}H-NMR Spectra of Compounds (N_1, N_2, N_6, N_9) in $CDCl_3$ Relative to TMS

Comp.code	Chemical Shift δ ppm
N_1	singlet in 5.30 (2H, O-CH_2), singlet in 10.01 (H, N-CH), multiplet 7.06-7.98 (12H,Aromatic protons)
N_2	singlet in 2.63 (3H, CH_3), singlet in 5.31 (2H,O-CH_2), singlet in 9.96 (1H,N-CH), 6.97-7.83 (12H,Aromatic protons)
N_6	multiplet in 6.61-7.76 (13H, Aromatic proton),doublet in 5.50(2H,2C-CH=),doublet in 6.00 (1H, Aryl-CH=), singlet in 3.44 (2H, O-CH_2), singlet in 2.52 (3H,-CH_3).
N_9	multiplet in 6.80-7.88 13H, Aromatic proton), doublet in 4.95 (1H, C-CH=), doublet in 5.52 (1H, Aryl-CH=), singlet in 4.03 (2H, O-CH_2), singlet in 2.60 (3H,=C-CH_3), singlet in 3.10 (3H, N-CH_3).

Table 11: The ^{13}C-NMR Spectra of Compounds (N_4, N_5) in $CDCl_3$ Relative to DMSO

Comp.code	Chemical Shift δ ppm
N_4	129.98-134.56 (13H, Aromatic carbons), 40.58 (2H, O-CH_2), 32.19 (3H,=C-CH_3), 35.46 (3H, N-CH_3), 120.11 (0H, CO-C=), 127.88 (0H, N-C=), 165.44 (0H, N-CO in antipyrine ring), 170.22 (0H, N-CO in seven heterocyclic ring) 108.44, 140.09 (1H, N-CH-O).
N_5	21.11 (3H, CH_3), 69.69 (2H, O-CH_2), 145.94 (1H, N-CH), 161.76 (0H, N-CO), 117.25-136.95 (12H, Aromatic Carbons).

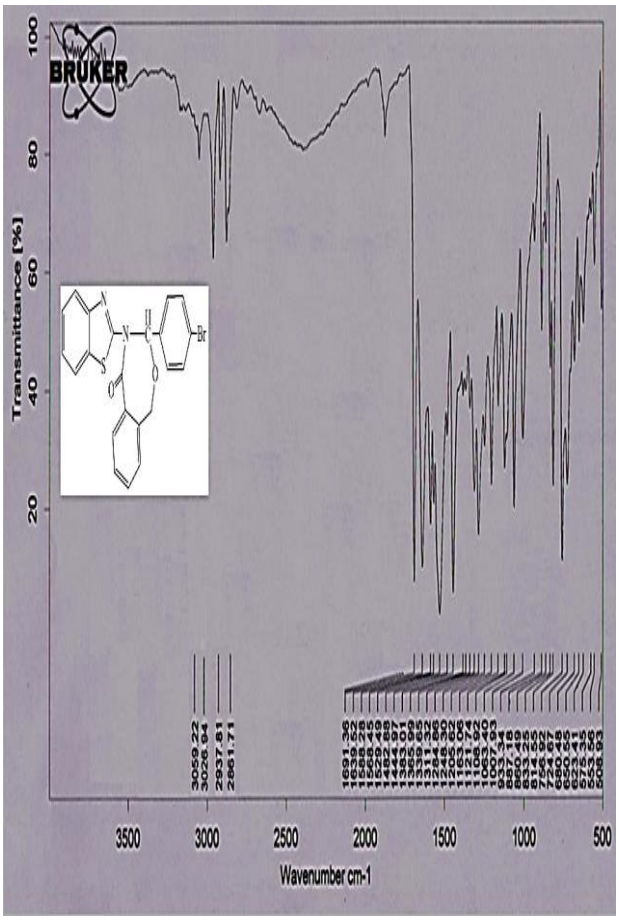


Figure (3) FT-IR spectra of N1

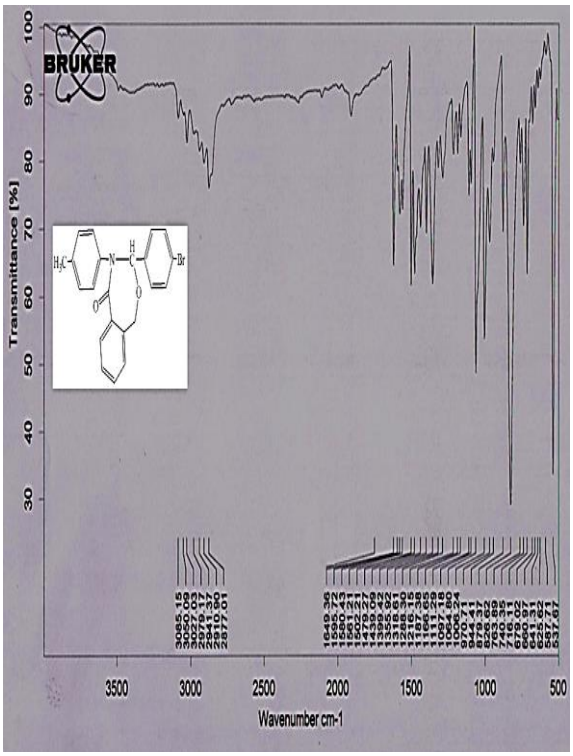


Figure (4) FT-IR spectra of N2

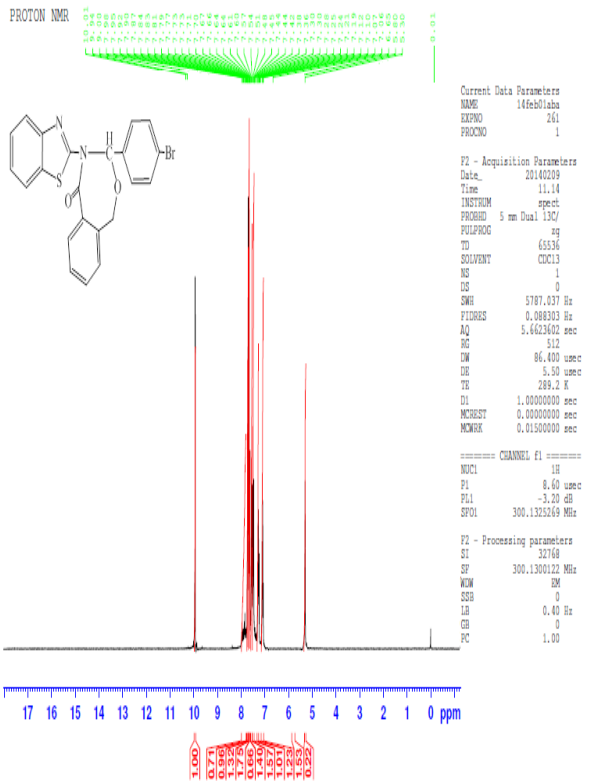


Figure (5) 1H-NMR Spectra of N1

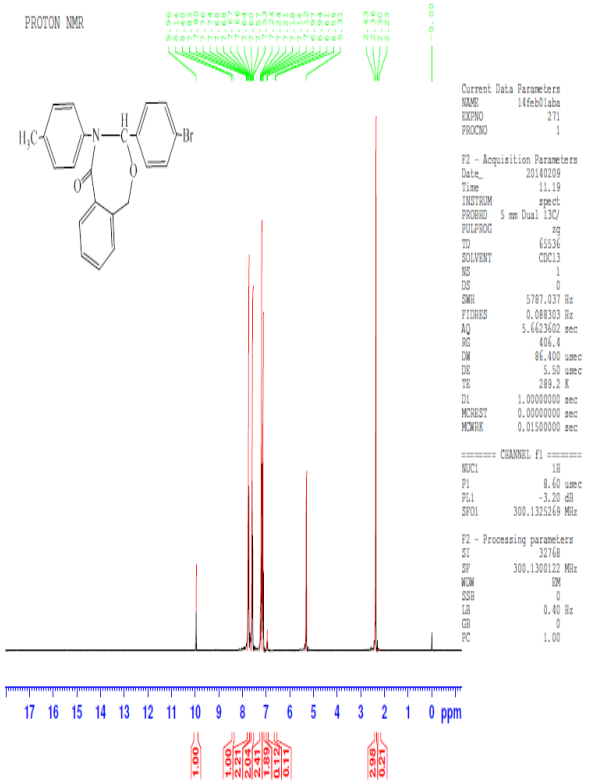


Figure (6) 1H-NMR Spectra of N2

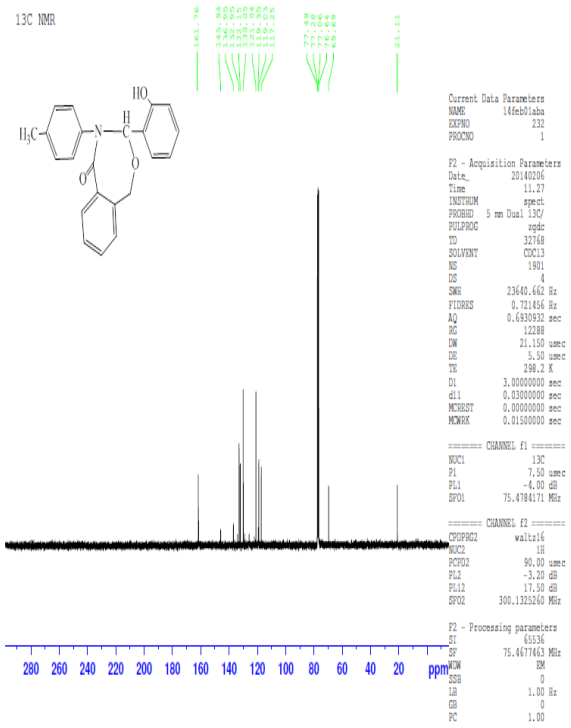


Figure (7) ^{13}C-NMR Spectra of N_5

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تصنيع وتوصيف مشتقات جديدة لـ1,3-oxazepin-4-ones من تفاعل قواعد شف مع Phthalide

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الخلاصة:

تم تحضير عدد من المشتقات الجديدة لمركبات 1,3–oxazepin–4–ones derivatives من تفاعل الاضافة–الحلقية المحفزة بالحامض لقواعد شف مع الفثالايد بالتصعيد في رباعي هايدروفيوران تحت ظروف جافة وبمنتوج عالي. حضرت قواعد شف من تفاعل الالديهايدات والكيتونات والجالكونات الاروماتية المحضرة مع الامينات الاروماتية الاولية. وقد شخصت النواتج بواسطة درجات انصهارها واطياف FT–IR ، UV–Vis، ^{1}H–NMR و ^{13}C–NMR.