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Synthesis and Characterization of Novel 1,3,4,9a-Tetrahydrobenzo[*e*][1,3]oxazepin-5(5a*H*)-one Derivatives via Cycloaddition Reactions of Schiff Bases

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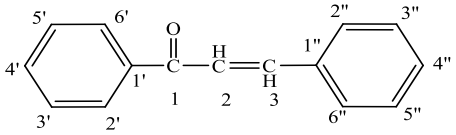
ABSTRACT

A series of novel 1,3,4,9a-tetrahydrobenzo[*e*][1,3]oxazepin-5(5a*H*)-one derivatives were synthesized by the reaction of Schiff bases with bicyclo[2.2.2]oct.7-ene-2,3,5,6-tetracarboxylic anhydride in anhydrous acetonitrile under dry and reflux conditions with high yields via polar cycloaddition. Schiff bases were synthesized by the reaction of aromatic aldehydes, ketones or prepared chalcones with primary aromatic amines. The products were identified by their melting point, FT-IR, UV-Vis-spectra, ^{1}H-NMR and^{13}C-NMR spectra.

Introduction

Chalcones are class of naturally occurring compounds of great importance as precursors and key intermediates for organic and bio-organic synthesis and as optical materials, UV-absorbing filters, holographic papers, liquid crystal components and food industry.[1-4] Synthesis of chalcones have been achieved by using various precursors and different methods, such as Cleisen-Schmidt, Friedel-Craft acylation, Suzuki coupling reaction Wittig reaction and Von-Konstanecki method.[5-8] 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.[9-10] The general structure of chalcone is represented by figure (1).



Figure(1): Chalcone structure.

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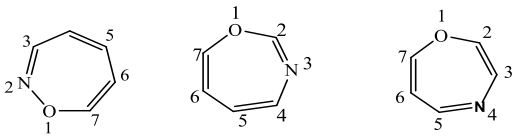
Chalcones possess a wide spectrum of biological activities and pharmacological applications such as anti-cancer, anti-bacterial, anti-microbial, anti-tubercular, anti-viral, anti-malaria, anti-leis mania, anti-ulcerative anti-oxidant, anti-hyperglycemic, anti-inflammatory, analgesic anti-diabetic[11-14] in addition to their uses as insecticides and pesticides.[15]

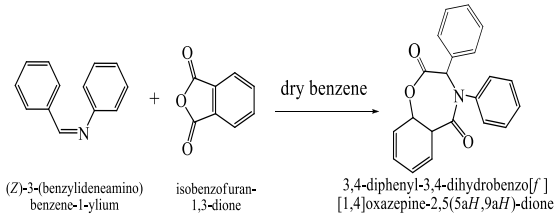
Schiff bases (imines) are class of compounds containing the azomethine group (C=N), originally prepared by by Hugo Schiff by the condensation of amino group in primary amines, amino acids and hydrazines with an active carbonyl group of aldehydes and ketones via azeotropic distillation with simultaneous removal of water.[16-18] Schiff bases are versatile precursors in the synthesis of organic, bio-organic, organometallic, heterocyclic and industrial compounds via ring closure, cycloaddition and replacement reactions[19]. Schiff' s bases exhibit a wide range of pharmacological activities like; anti-microbial[20], antiparasitic[21] anti-inflammatory[22], anti-cancer anti-tummor, anti-fungal,ant-leukemia activities.[23-27] In addition they have been used as a protective agents for natural rubber and amino groups in organic and bio organic synthesis [28].

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.[29]



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 others beside photochemical ring expansion reactions of pyrimidines and aziridines.(30)



They possess a wide range of biological activities and pharmacological applications such as anti-depressant, analgesic, psychoactive drug, anti-convulsant, enzyme inhibitor, anti-histaminic, anti-allergic, anti-bacterial, anti-fungal and anti-inflammatory, anti-tumor, anti-microbial, anti-oxidant, anti-corrosion.(31-34)

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 (R_1, R_2& R_3).

Chalcones were synthesized according to literature procedure.(5) 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. Schiff's bases were synthesized by literature procedure.(17)

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 twice form ethanol. The structural formuli, names, melting points, colors, and percentage yields for the synthesized Schiff bases are given in table1.

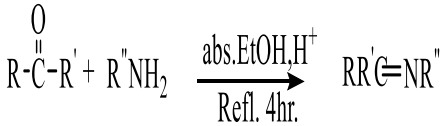
Synthesis of 1, 3, 4, 9a- tetrahydrobenzo [*e*] [1,3] oxazepin -5(5a*H*) – one derivatives (K_1-K_9)

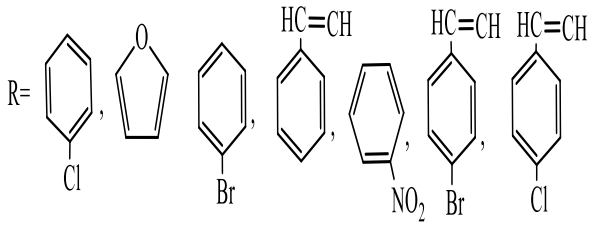
In well dried 100-ml round-bottom flask equipped with condenser and anhydrous calcium chloride tube guard a mixture of Schiff bases (0.01mol) and isobenzofuran-1(3*H*)-one (0.01mol) dissolved in (20ml) of tetrahydrofuran (THF) with trace of glacial acetic acid as catalyst was refluxed for 3hr and left 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 3,4-dihydrobenzo[*e*][1,3]oxazepin-5(1*H*)-one derivatives are given in table2.

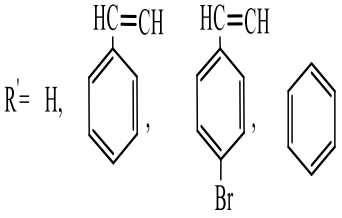
Results and Discussion

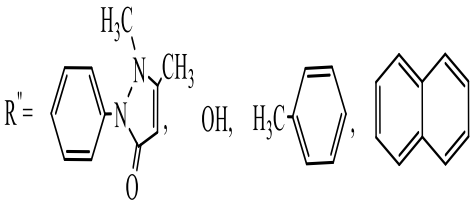
In this work the synthesis of novel 1,3-oxazepane-4,7-dione by direct reaction of several Schiff bases with bicyclo[2.2.2]oct-7-enes bicyclo [2.2.2]oct-2-ene in dry acetonitrile is reported. Chalcones were synthesized from commercially available aldehydes and ketones and identified by their melting points, FT-IR and UV-Vis. spectra, tables,(1),(4) and (7). Formation of the products were followed up by the appearance of the streching absorption bands of (C=O) group at (1652-1668)cm^{-1} and of (C-H aromatic) group at (3052-3082)cm^{-1} beside the characteristic bands of the residual groups of the structure in the FT-IR spectra, 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 (R_1-R_3).

Schiff bases were synthesized from commercially available aldehydes, ketones and primary amines and identified by their melting points, FT-IR and UV-Vis. spectra, tables,(2),(5) and (8). Formation of the products were followed up by the appearance of the streching absorption bands of azomethine (C=N) group at (1575-1654) cm^{-1} of the resulting imines beside the characteristic bands of the residual groups in the structure in the FT-IR spectra, table,(8). The UV-Vis. Spectra of these imines showed absorption maxima at (209-330)nm owing to the electronic transfers π-π* and n-π* characteristic of the structures of the synthesized imines (F_1-F_8).

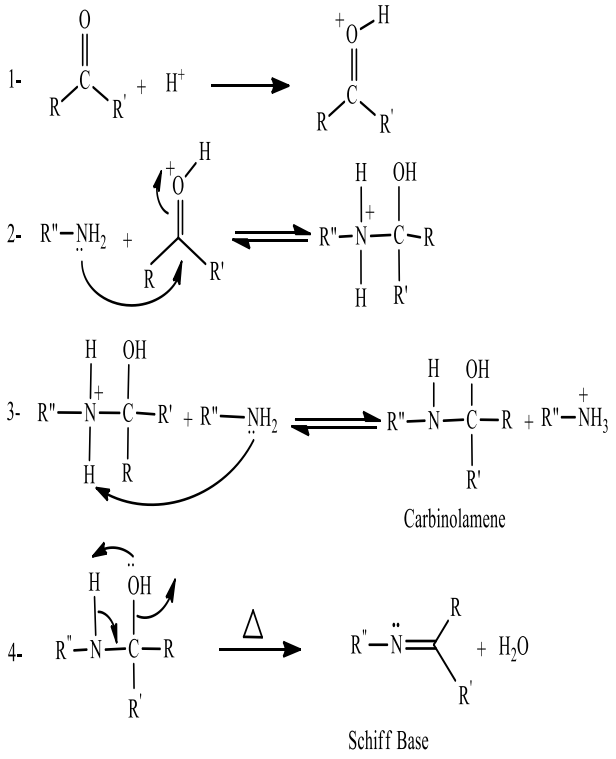








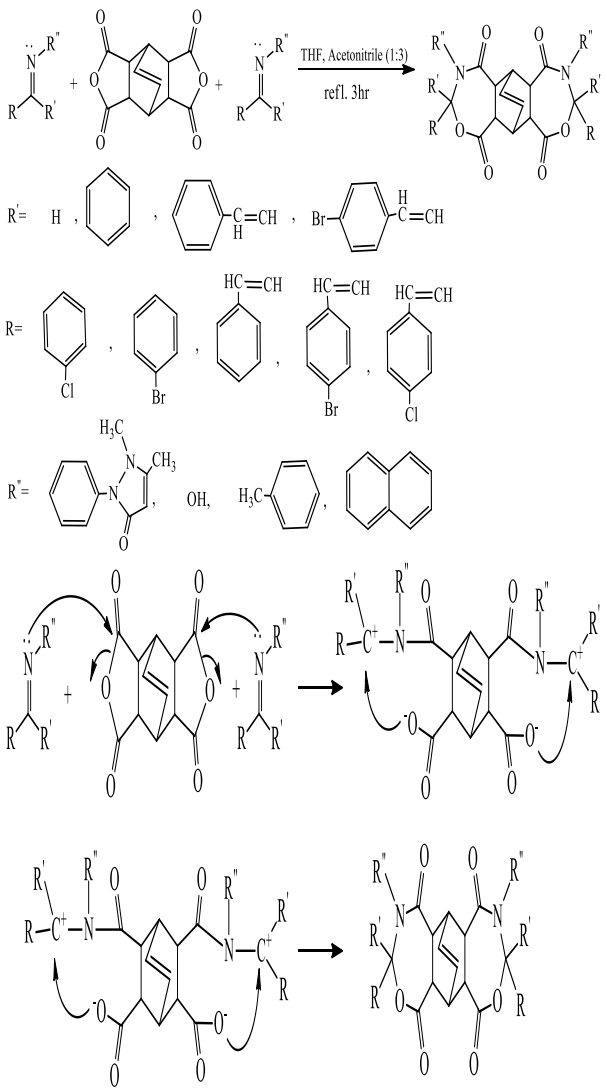
The mechanism of Schiff bases formation was thoroughly discussed and established as illustrated by scheme(1).[35-37]



Sceme (1): The plausible mechanism of formation of Schiff's bases.

The synthesis of novel 1,3,4,9a-tetrahydrobenzo [*e*] [1,3] oxazepin- 5(5a*H*) –one derivatives were achieved by the reaction of imines with bicyclo [2.2.2] oct-7-enes (bicycle [2.2.2] oct-2-ene and 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 (1679-1740)cm-1 and (1638-1704)cm^{-1} indicative of formation of C=O (lactone) and C=O (lactame) respectively, beside the characteristic bands of the residual groups in the structure, table,(9). Figures (3) and (4) showed the FT-IR spectra of M_3 and M_8. The UV-Vis. spectra show absorption maxima at (232-370)nm owing to the electronic transfers π-π* and n-π* characteristic of the structure of the synthesized 1,3,4,9a- tetrahydrobenzo [*e*][1,3] oxazepin -5(5a*H*)-one derivatives, table,(6). The ^{1}H-NMR spectrum of compound K_1 in solvent $CDCl_3$ showed chemical Shifts, δ(ppm), multiplet at 7.11-7.80 (18H, Aromatic protons), singlet at 8.98 (2H, 2N-CH-O), triplet at 5.00 (2H, 2NCO-CH), triplet at 5.87 (2H, 2OCO-CH), triplet at 8.13 (2H,-CH=CH), quartet at 6.77 (2H, 2CH-C=), singlet at 2.00 (6H,2=C-CH_3), singlet at 2.61 (6H, 2N-CH_3). The spectrum of compound K_3 showed chemical shifts, δ(ppm) at: multiplet at 7.66-7.94 (16H, Aromatic

protons), singlet at 2.55 (6H, 2 $\underline{CH_3}$), triplet at 5.83 (2H, 2NCO-$\underline{CH}$), triplet at 6.11 (2H, 2OCO-$\underline{CH}$),triplet at 9.96 (2H, 2 N-$\underline{CH}$-O), quartet at 6.88 (2H, 2 $\underline{CH}$-C=), triplet at 8.73 (2H,-$\underline{CH=CH}$), other chemical shifts, δ(ppm) in table (10), figure(5) showed chemical shifts of K_3. The ^{13}C-NMR spectrum of compound K_8 in solvent $CDCl_3$ showed chemical shifts, δ(ppm), 20.41 (6H, 2 -$\underline{CH_3}$), 54.48 (2H, 2 NCO$\underline{CH}$), 69.74 (2H, 2 OCO$\underline{CH}$), 46.22 (2H, 2 $\underline{CH}$-C=C) 54.48 (0H, 2 N-$\underline{C}$-O), 114.08 (2H, 2 O-C-$\underline{CH}$=), 122.52 (2H, 2 =$\underline{CH}$-Aryl), 141.73 (2H, 2 $\underline{CH}$=), 144.43 (0H, 2N-C-O), 199.74 (0H, 2O-$\underline{CO}$), 190.30 (0H, 2N-$\underline{CO}$), 127.35-136.62 (26H, Aromatic carbons), other chemical shifts, δ(ppm) in table (11), figure (6) showed chemical shifts of K_8. The sequence of the reaction is given by scheme (2):



Scheme(2): The sequence of the reaction and the suggested mechanism of imine with bicycle [2.2.2] oct-7-enes bicyclo [2.2.2]oct-2-ene.

It may be concluded that the reaction takes place via concerted dipolar cycloaddition mechanism as given by scheme (2). The nucleophilic imine attacked the electrophilic carbon atom of the carbonyl group to give dipolar intermediate in the first step. Whereas the, second step include the collapses of the intermediate to give the target molecule.

Table1. Structural formulas, names, melting points, colors, and % yields of Chalcones (R_1-R_3).

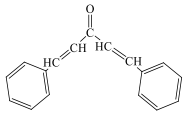
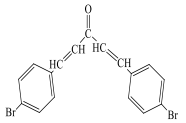
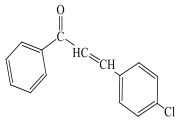
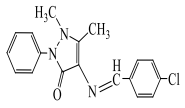
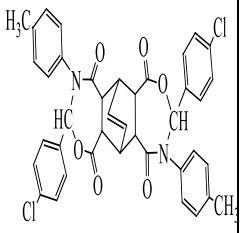
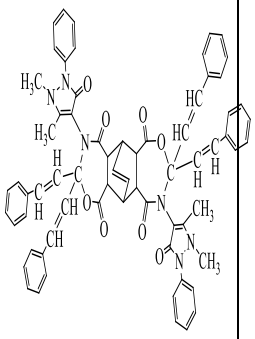
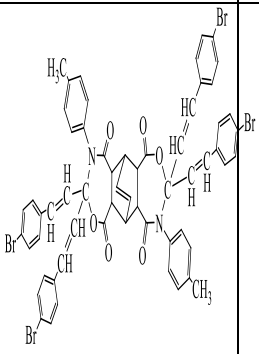
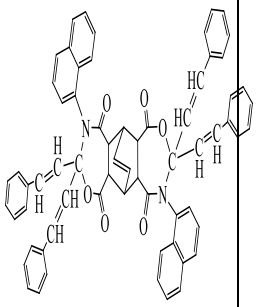
Comp. No.	Structural formula	Name	Yield %	m.p.°C	Color
R_1		1,5-diphenyl penta -1,4-dien-3-one	85%	92-95	yellow
R_2		1,5-bis(4-bromophenyl) penta-1,4-dien-3-one	65%	192-195	yellow
R_3		3-(4-chlorophenyl) -1-phenylprop-2-en-1-one	87%	112-115	yellow

Table2. Structural formulas, names, melting points, colors, and % yields of Schiff bases (F_1-F_8).

Comp. No.	Structural formula	Name	Yield %	m.p. °C	Color
F_1		4-(4-chlorobenz ylid eneamino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one	82%	254-256	Bright Pale Yellow

Comp. No.	K_1	K_2	K_3	F_2	F_3	F_4	F_5	F_6	F_7	F_8
Structural formula										
Name	3,9-bis(4-chloro Phenyl)-4,8-bis(1,5-dimethyl-3-ox o-2-ph enyl-2,3-dihydro-1 H-pyrazol-4-yl)-3,4 ,6, 6a,8,9,12,12a-oct ahydro -6,12-etheno benzo[1,2-e:5,4-e']bi s ([1,3]oxazep ine)-1, 5 ,7,11(5aH,11aH) -traone	3,9-bis(4-chlorophenyl)-4,8-dihydrox y -3,4,6,6a ,8,9,12,12a -octahydro-6,12-eth enobenzo[1,2-e:5, 4-e']bis([1,3]oxazep ine)-1,5,7, 11(5aH,11 aH)- tetraone	(3R,12R)-3,9-bis(4-brom ophenyl)-4,8-di-p-tolyl-3, 4,6,6a,8 ,9,12,12a-octahy dro -6,12-ethenobenzo[1 ,2-e:5,4-e']bis([1,3]oxaze pine)-1,5,7,11(5aH,11aH)-tetraone	4-chlorobenzald ehyde oxime	*N*-(4-bromobenzylid ene)-4-methylaniline	*N*-(4-chlorobenzylid ene)-4-methylaniline	4-(1,5-diphenylpenta -1,4-dien-3-ylidenea mino)-1,5-dimethyl-2-phenyl-1*H*-pyrazol -3(2*H*)-one	*N*-(1,5-bis(4-bromop henyl)penta-1,4-ien-3-ylidene)-4-methyla niline	*N*-(1,5-diphenylpen ta-1,4-dien-3-ylidene)naphthalen-1-amine	*N*-(3-(4-chlorophenyl)-1-phenylallylidene) -4-methylaniline
Yield %	83%	85%	85%	83%	92%	93%	59%	72%	71%	75%
m.p. °C	248-250	199-201	134-136	59-60						
Color	Bright pale yellow	Pale pink	Bright white	Bright white	Bright white	Bright white	Pale orang	Bright light tan	Brown	Light tan

Table3. Structural formulas, names, melting points, colors, and % yields of 1,3,4,9a-tetrahydrobenzo[*e*][1,3]oxazepin-5(5a*H*)–one derivatives (K_1-K_8).

K4		**3,9-bis(4-chlorophenyl)-4 ,8-di-p-tolyl-3,4,6,6a,8,9, 12,12a-octahydro-6,12-et henobenzo[1,2-e: 5,4-e'] bis([1,3]oxazepine)-1,5,7, 11(5aH,11a H)-tetraone**
K5		**4,8-bis(1,5-imethyl-3-oxo -2-phenyl-2,3-dihydro-1 H-pyrazol -4-yl)-3,3,9,9-tetra styryl-3,4,6,6a,8,9, 12,12a-octahydro -6,12-ethenobenzo [1,2-e:5,4-e']bis ([1, 3]oxazepine)-1,5, 7,11 (5aH,11aH)- tetraone**
K6		**(3S,12R)-3-((E)-4-bromo styryl)-3,9,9-t ris(4-brom ostyryl)-4,8-di-p-tolyl-3,4 ,6,6 a,8,9,12,12a- octah ydro-6,12-ethenob enzo [1,2-e:5,4-']bis([1,3]oxa zepine)-1, 5,7, 11(5aH, 11aH)-tetraone**
K7		**(12S)-4,8-i(naphtha l en-1-yl)-3-((E)-sty ryl)-3,9,9-tristyryl-3,4,6,6a,8,9,12, 12a-octahydro-6,12-ethe nobenzo[1,2-e:5,4-e']bis ([1,3]oxazepin e)-1,5,7,11 (5aH,11 aH)-tetraone**

K4	**78%**	**242-245**	**Bright pale yellow**
K5	**86%**	**90-92**	**Bright pale nutty**
K6	**88%**	**206-208**	**Bright pale yellow**
K7	**81%**	**109-110**	**Bright nutty**

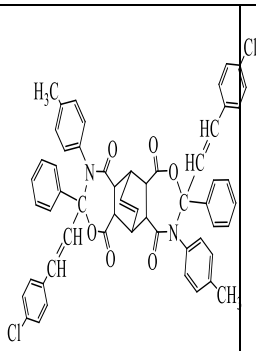
K8		**(12S)-3-((Z)-4-chlorostyr yl)-9-(4-chlorostyryl)-3,9-diph enyl-4,8-di-p-tolyl-3,4,6,6a,8,9,12,12a-octah ydro-6,12-eth enobenzo [1,2-e:5, 4-e']bis([1,3]oxa zepine)-1,5,7,11(5aH,11 aH)-tetraone**	**91%**	**96-98**	**Bright pale yellow**

Table 4: The UV-Visible Absorption Max (nm) in DMSO of chalcones (R_1-R_3).

Comp. code	Wave Length: λ/nm	
R_1	234.0	350.0
R_2	228.0	344.0
R_3	263.0	312.0

Table 5: The UV-Visible Absorption Max (nm) in Ethanol of Schiff Bases (F_1-F_8)

Comp. code	Wave Length: λ/nm	
F_1	215.0	272.0
F_2	235.0	330.0
F_3	225.0	270.0
F_4	244.0	310.0
F_5	209.0	284.0
F_6	230.0	270.0
F_7	225.0	305.0
F_8	228.0	290.0

Table 6: The UV-Visible Absorption Max (nm) in chloroform of 1,3,4,9a-Tetrahydrobenzo [*e*][1,3]oxazepin-5(5a*H*)-one derivatives (K_1-K_8)

Comp. Code	Wave Length: λ/nm	
K_1	232.0	350.0
K_2	245.0	368.0
K_3	265.0	355.0
K_4	240.0	360.0
K_5	240.0	352.0
K_6	248.0	335.0
K_7	265.0	370.0
K_8	246.0	354.0

Table7. FT-IR of Chalcones (R_1-R_3).

Comp. No	IR(KBr), ν(cm^{-1})					
	C=O Alkene	C=C Aromatic	C=C Aromatic	C-H aromatic	C-H alkene	Others
R_1	1652	1595	1573	3082	3100	--

R_3	R_2
1668	1656
1576	1600
1569	1581
3075	3052
3086	3095
C-Cl 762	C-Br 536

Table8. FT-IR of Schiff bases (F1-F8).

Comp. No	IR(KBr), ν(cm^{-1})								
	C=C Arom	C=N	C-H Eli.	C-H Arom	C-N	=C-H Alke	C=CAlke	C-Cl	Others
F_1	1580	1575	--	3035	1165	3067	--	754	O-H3342b,N-O1330
F_2	1460	1655	2890	3105	1169	3110	--	779	N-N 1144
H_3	1498	1616	2934	3095	1167	3115	--	--	--
F_4	1566	1641	2899	3071	1139	3092	--	741	--
F_5	1566	1652	2976	3102	1142	3127	1592	--	N-N 1142
F_6	1512	1645	2967	3074	1167	3091	1564	--	C-Br 579
F_7	1463	1592	--	3083	1157	3098	1530	--	--
F_8	1485	1611	2910	3099	1152	3115	1554	756	--

Table9. FT-IR of 1,3,4,9a-tetrahydrobenzo [*e*][1,3]oxazepin-5(5a*H*)–one derivatives (K_1-K_8)

Comp. No	FT-IR(KBr), ν(cm^{-1})
C-O	
C=C Arom.	
C=C alkene	
C-H Aro.	
C-H Ali.	
C-N	
C=O Lactam	
C=O Lacton	
Others	

K_1	K_2	K_3	K_4	K_5	K_6	K_7	K_8
1215	1260	1235	1239	1223	1247	1250	1216
1610	1575	1561	1592	1608	1598	1588	1519
1624	1588	1597	1635	1614	1615	1606	1591
3100	3040	3083	3078	3090	3078	3092	3021
2920	2855	2972	2892	2924	2897	2876	2917
1130	1205	1186	1194	1130	1167	1130	1182
1685	1630	1704	1600	1671	1675	1638	1659
1740	1695	1775	1699	1710	1716	1723	1679
C-Cl 711	C-Cl 698,OH3406	C-Br 585	C-Cl 729	C-H alkene 3110 N-N 1150	C-H alkene 3116 C-Br 544	C-H alkene 3115	C-H alkene 3095 C-Cl 732

Table 10: The ^{1}H-NMR Spectra of Compounds (K_1, K_3, K_4) in $CDCl_3$ Relative to TMS

Comp.	Chemical Shift δ ppm
K_1	multiplet in 7.11-7.80 (18H, Aromatic protons), singlet in 8.98 (2H, 2N-CH-O), triplet in 5.00 (2H, 2NCO-CH), tiplet in 5.87 (2H, 2OCO-CH), triplet in 8.13 (2H,-CH=CH), quartet in 6.77 (2H, 2CH-C=), singlet in 2.00 (6H,2=C-CH_3), singlet in 2.61 (6H, 2N-CH_3)
K_3	multiplet in 7.66-7.94 (16H, Aromatic protons), singlet in 2.55 (6H, 2 CH_3), triplet in 5.83 (2H, 2NCO-CH), triplet in 6.11 (2H, 2OCO-CH),triplet in 9.96 (2H, 2 N-CH-O), quartet in 6.88 (2H, 2 CH-C=), triplet in 8.73 (2H,-CH=CH)
K_4	Singlet in 9.37 (2H, 2N-CH-O), triplet in 8.33 (2H, 2CH=CH), quartet in 6.22

	(CH-C=), triplet in 5.02 (2H, 2NCO-CH), triplet in 5.91 (2H, 2OCO-CH), singlet in 2.72 (6H,2CH_3), multiplet in 7.10-7.70 (8H, Aromatic protons).

Table 11: The ^{13}C-NMR Spectra of Compounds (K_2, K_8) in Acetic Relative to DMSO

Comp.	Chemical Shift δ ppm
K_2	**60.33 (2H, 2 NCOCH), 72.25 (2H, 2 OCOCH), 50.22 (2H, 2 CH-C=C), 142.55 (0H, 2 N-CH-O), 141.88 (2H, 2 CH=), 183.21 (0H, 2 O-CO), 173.26 (0H, 2 N-CO), 129.30-140.02 (6H, Aromatic carbons).**
K_8	**20.41 (6H, 2 -CH_3), 54.48 (2H, 2 NCOCH), 69.74 (2H, 2 OCOCH), 46.22 (2H, 2 CH-C=C), 54.48 (0H, 2 N-C-O), 114.08 (2H, 2 O-C-CH=), 122.52 (2H, 2 =CH-Aryl), 141.73 (2H, 2 CH=), 144.43 (0H, 2N-C-O), 199.74 (0H, 2 O-CO), 190.30 (0H, 2 N-CO), 127.35-136.62 (26H, Aromatic carbons)**

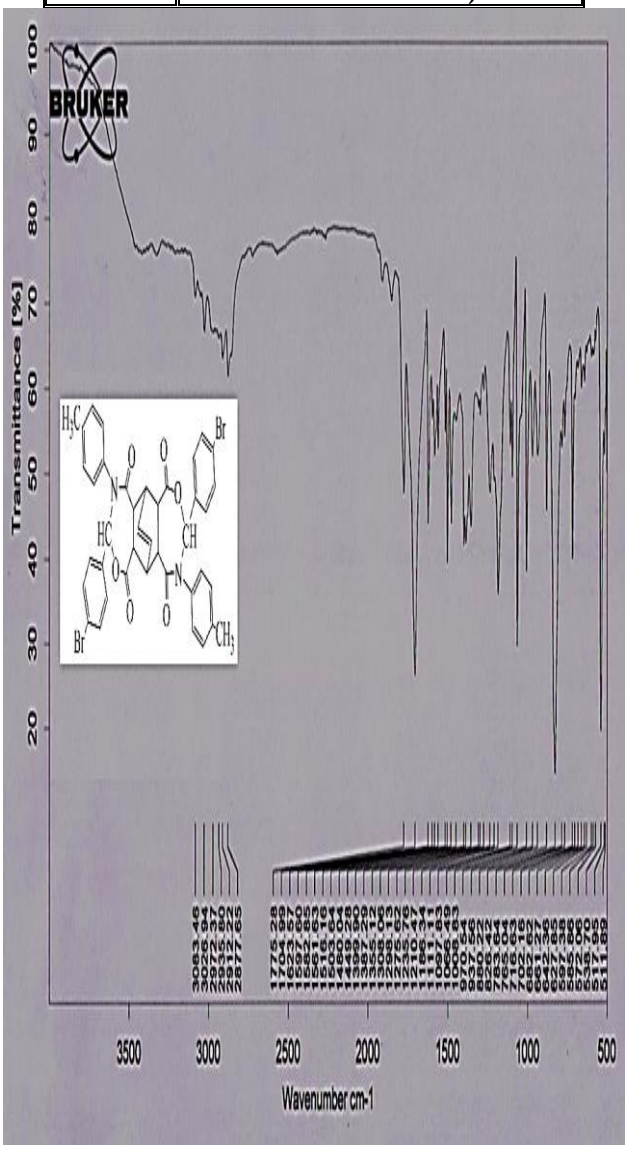


Figure (3) FT-IR spectra of K_3

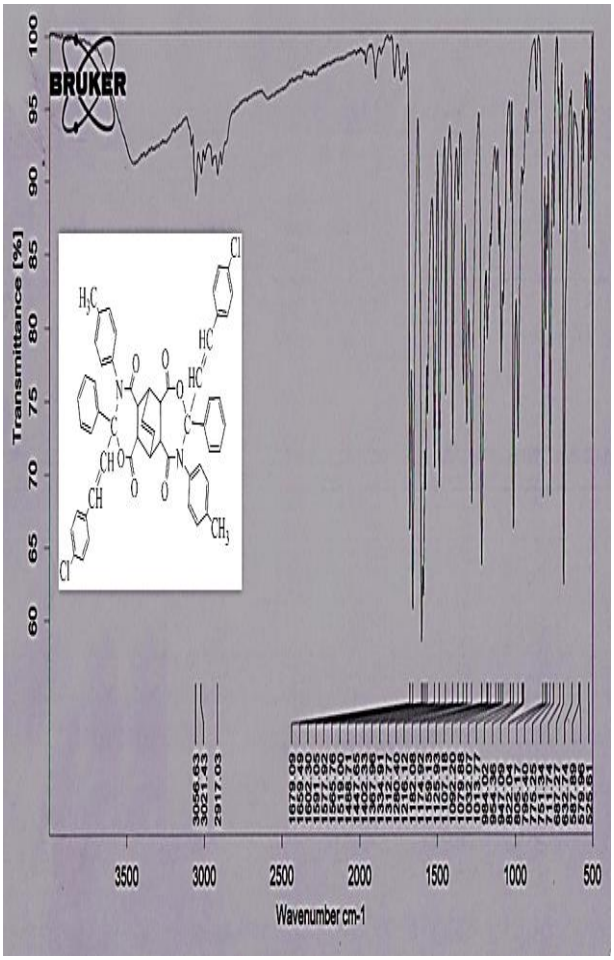


Figure (4) FT-IR spectra of K_8

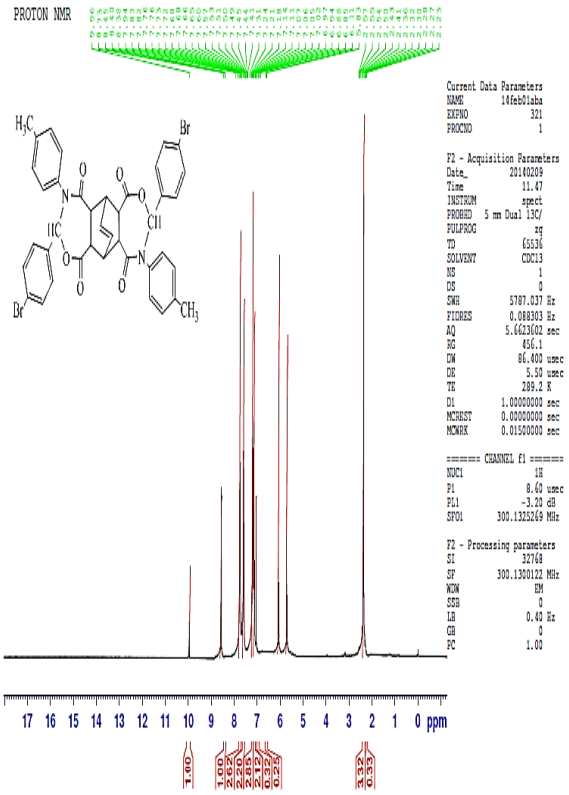


Figure (5) ^{1}H-NMR Spectra of K_3

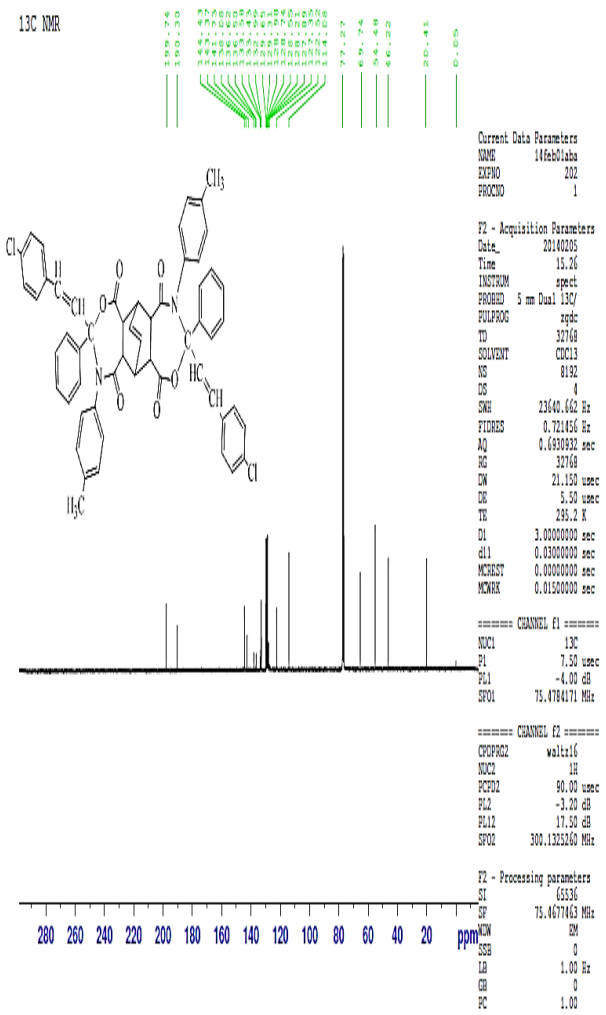


Figure (6) ^{13}C-NMR Spectra of K_8

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تحضير وتوصيف 1,3,4,9a-Tetrahydrobenzo[e][1,3]oxazepin-5(5aH)-one Derivatives باستعمال تفاعل الاضافة الحلقية لقواعد شف

عبيد حسن عبد راسم فراج مسلم خالد محمود محمد

الخلاصة:

حضرت مشتقات جديدة لمركبات 1,3,4,9a-Tetrahydrobenzo[*e*][1,3]oxazepin-5(5a*H*)-one, من تفاعل قواعد شف مع الانهدريد الحلقي الثنائي [2.2.2.] أوكت 7- اين-2، 3، 5، 6-رباعي كاربوكسيل انهدرايد بتصعيدها في مذيب الاسيتونترايل الجاف تحت ظروف جافة بمنتوج عالي. حضرت قواعد شف من تفاعل الالديهايدات والكيتونات والجالكونات المحضرة الاروماتية مع الامينات الاروماتية الاولية. شخصت النواتج بواسطة درجات انصهارها واطياف FT-IR, UV-Vis, ^{1}H-NMR و ^{13}C-NMR.