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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

**Obaid H. Abid*** **Rasim F. Muslim**** **Khalid M. Mohammed*****

* University of Fallujah
** University of Anbar - College of Applied Sciences-Heet,
*** University of Tikrit - College of Education for Pure Sciences

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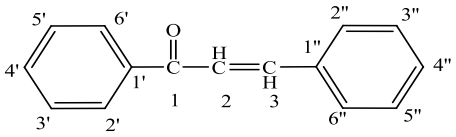
**ABSTRACT**

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).



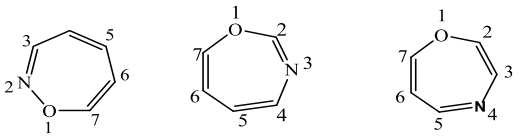

Figure(1): Chalcone structure.

* Corresponding author at University of Fallujah: .E-mail address:

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]

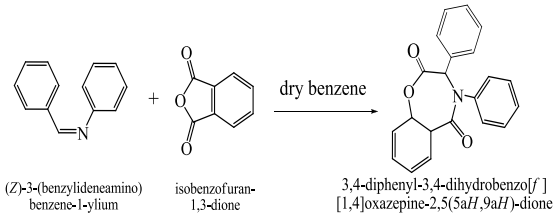
Schiff bases (imines) are class of compounds containing the azomethine group (C=N), originally prepared by by Hugo Schiff by the condensation of amino group in primary amines, amino acids and hydrazines with an active carbonyl group of aldehydes and ketones via azeotropic distillation with simultaneous 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.

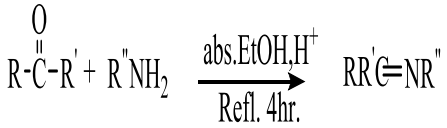
## Synthesis of 1, 3, 4, 9a- tetrahydrobenzo [*e*] [1,3] oxazepin -5(5a*H*) – one derivatives ($K_1$-$K_9$)

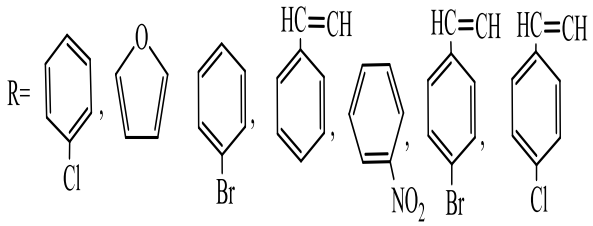
In well dried 100-ml round-bottom flask equipped with condenser and anhydrous calcium chloride tube guard a mixture of Schiff bases (0.01mol) and isobenzofuran-1(3*H*)-one (0.01mol) dissolved in (20ml) of tetrahydrofuran (THF) with trace of glacial acetic acid as catalyst was refluxed for 3hr and left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

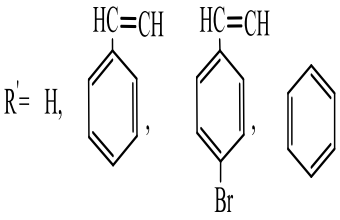
## Results and Discussion

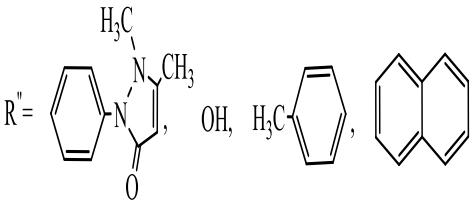
In this work the synthesis of novel 1,3-oxazepane-4,7-dione by direct reaction of several Schiff bases with bicyclo[2.2.2]oct-7-enes bicyclo [2.2.2]oct-2-ene in dry acetonitrile is reported. Chalcones were synthesized from commercially available aldehydes and ketones and identified by their melting points, 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 ($F_1$-$F_8$).

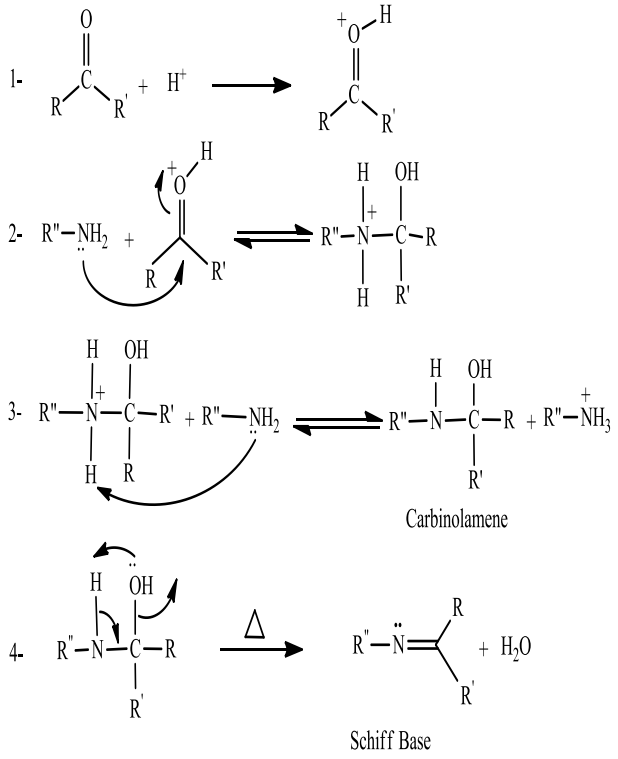






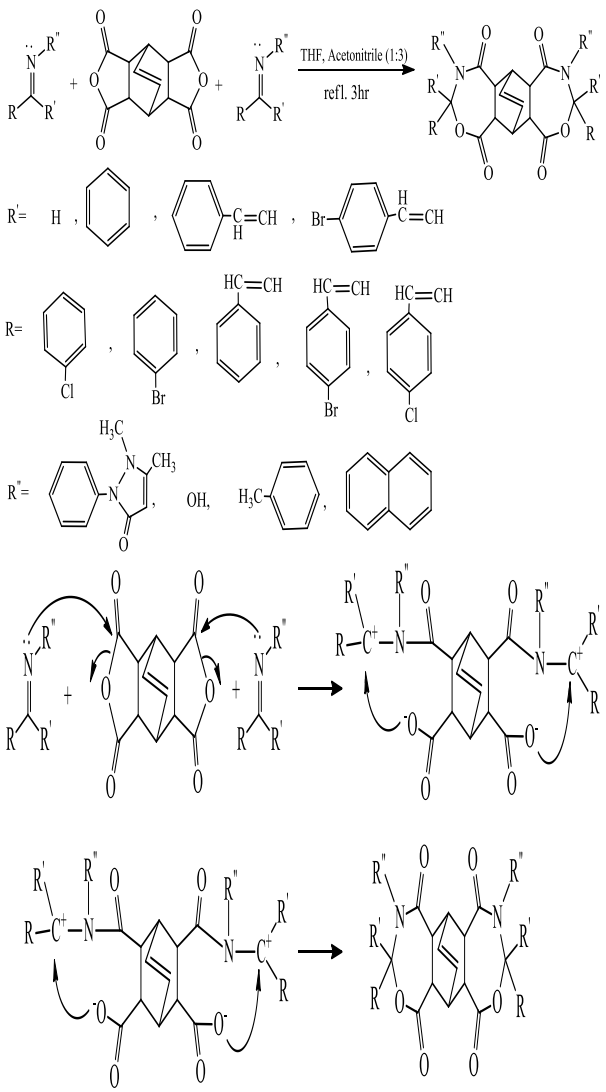
The mechanism of Schiff bases formation was thoroughly discussed and established as illustrated by scheme(1).[35-37]




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 ($R_1$-$R_3$).**

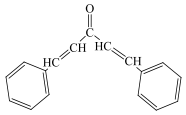
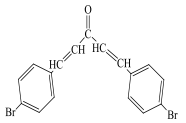
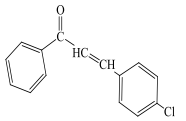
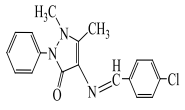
| Comp. No. | Structural formula | Name | Yield % | m.p.°C | Color |
|---|---|---|---|---|---|
| $R_1$ |  | 1,5-diphenyl penta -1,4-dien-3-one | 85% | 92-95 | yellow |
| $R_2$ |  | 1,5-bis(4-bromophenyl) penta-1,4-dien-3-one | 65% | 192-195 | yellow |
| $R_3$ |  | 3-(4-chlorophenyl) -1-phenylprop-2-en-1-one | 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. | $K_1$ | $K_2$ | $K_3$ |
|---|---|---|---|
| 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 |
| Yield % | 83% | 85% | 85% |
| m.p. °C | 248-250 | 199-201 | 134-136 |
| Color | Bright pale yellow | Pale pink | Bright white |

| Comp. No. | $F_2$ | $F_3$ | $F_4$ | $F_5$ | $F_6$ | $F_7$ | $F_8$ |
|---|---|---|---|---|---|---|---|
| Structural formula |  |  |  |  |  |  |  |
| Name | 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% | 92% | 93% | 59% | 72% | 71% | 75% |
| m.p. °C | 59-60 | 140-142 | 134-137 | | | | |
| Color | 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 ($K_1$-$K_8$).**

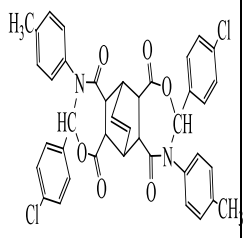
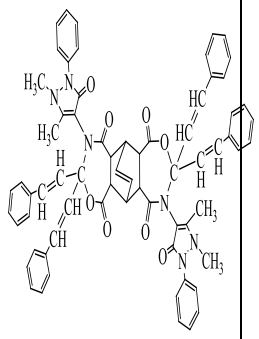
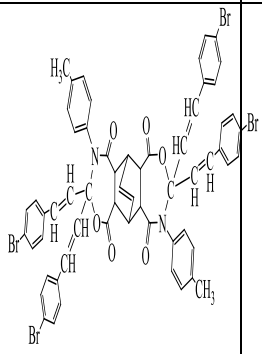
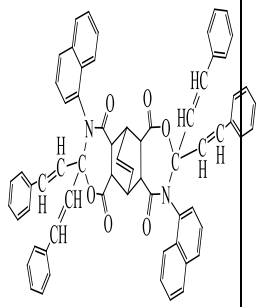
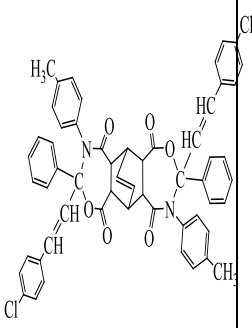
| | | | | | |
|---|---|---|---|---|---|
| **K4** |  | **3,9-bis(4-chlorophenyl)-4,8-di-p-tolyl-3,4,6,6a,8,9, 12,12a-octahydro-6,12-ethenobenzo[1,2-e: 5,4-e'] bis([1,3]oxazepine)-1,5,7, 11(5aH,11a H)-tetraone** | **78%** | **242-245** | **Bright pale yellow** |
| **K5** |  | **4,8-bis(1,5-imethyl-3-oxo -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** | **86%** | **90-92** | **Bright pale nutty** |
| **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** | **88%** | **206-208** | **Bright pale yellow** |
| **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** | **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,2$CH_3$), 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)** |

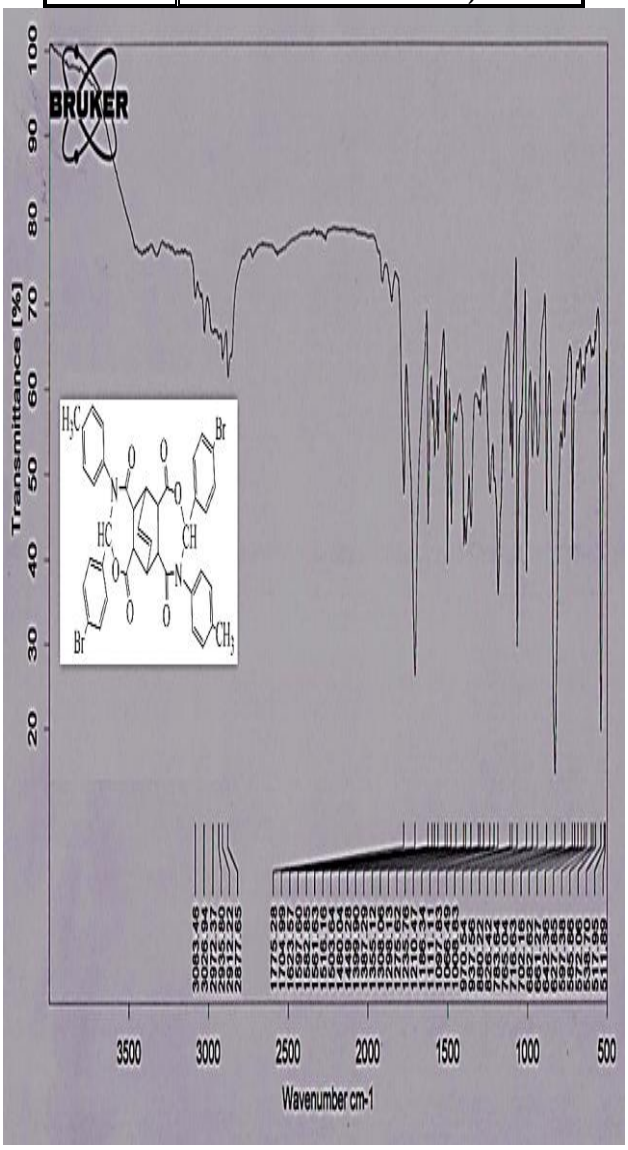



**Figure (3) FT-IR spectra of $K_3$**

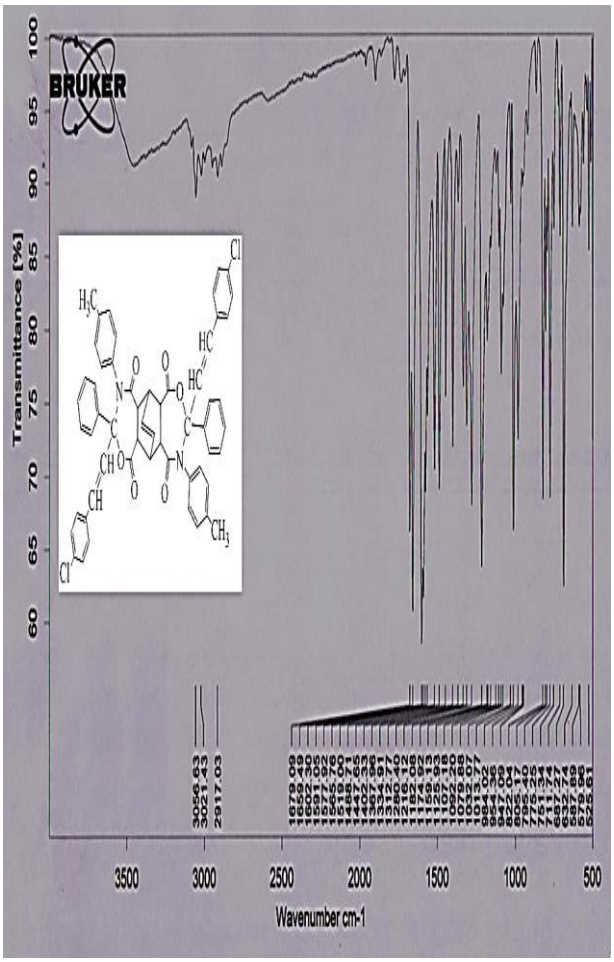



**Figure (4) FT-IR spectra of $K_8$**

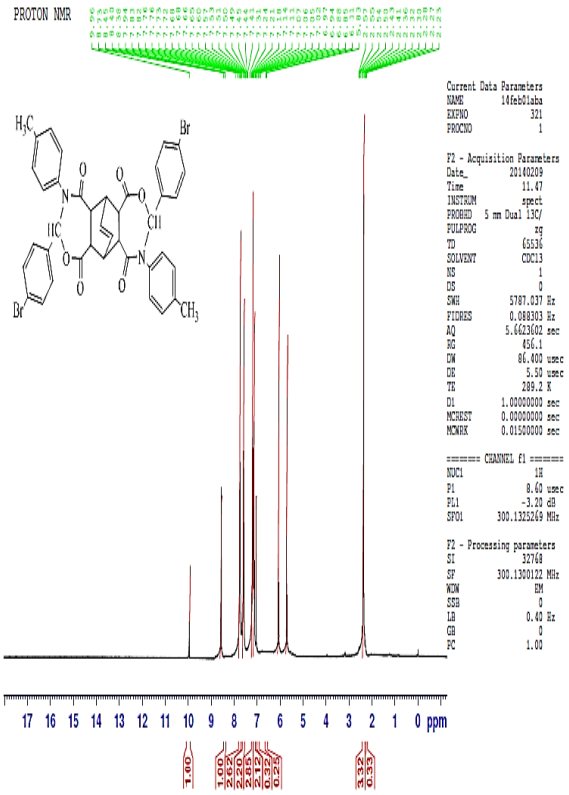



**Figure (5) $^1$H-NMR Spectra of $K_3$**

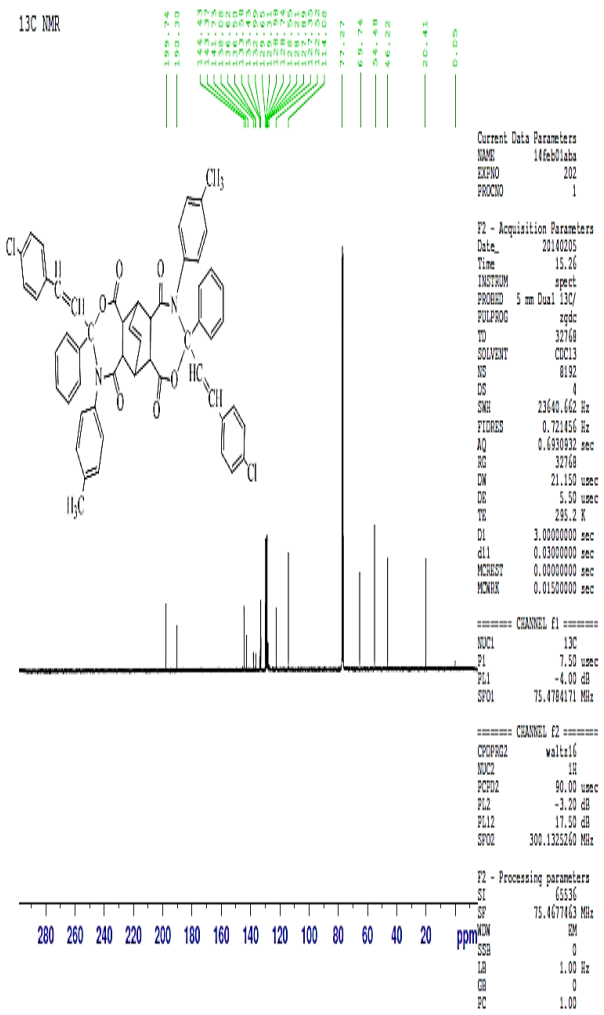



**Figure (6) $^{13}$C-NMR Spectra of $K_8$**

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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.