# Synthesis, mercuration, and halogenation of some furyl α,β-unsaturated ketons and study of biological activity

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#### Abstract

The first part of the research includes the preparation of some a, \beta-carbonyl compounds containing furan ring in a basic medium, compounds(1-4), also some difurylidine and furylidine-benzylidine compounds have been prepared from furfural and substituted benzaldehydes and cyclohexanone, compounds(5-9) in basic medium . The second part of the research included the mercuration of The resulting compounds with mercury (II) chloride compounds(10-19) followed by symmeterisation with NaI to afford compounds of the form (Fur)<sub>2</sub>Hg (20-28). Some of the mercurated compounds have been halogenated via the replacement of -HgCl by the reaction with iodine, compounds (29-31). The third part of the research included the biological activity of two of the prepared compounds (3,4). All of the prepared compounds have been identified by their melting points and IR and UV spectra and some of them by CHNS analysis and NMR techniques.

#### Introduction

 $\alpha,\beta$ -unsaturated ketones are important type of carbonyl compounds with the general structure:

## -c=c-c=o

In these compounds the carbonyl is conjugated with a carbon–carbon double bond and the double bond here is deactivated towards the electrophilic attack since the carbonyl groups tends to withdraw the electrons away from the double bond.

The most important derivatives of these compounds are the chalcones and the diarylidine acetone compounds  $^{(1,2)}$ . Chalcones are a class of important compounds in medicinal and pharmaceutical fields including: Antimalarial and antileishmanial <sup>(3)</sup>, antibacterial <sup>(4)</sup>, antipyretic <sup>(5)</sup>, anticarcinogenic <sup>(6)</sup> and anti-inflammatory <sup>(7)</sup>. In the present work some  $\alpha,\beta$ unsaturated compounds and diarylidine acetone compounds containing furan ring have been prepared and one of the important reaction is aromatic electrophilic substitution which was carried out on them is the mercuration via mercury (II)chloride (mercuration). Heterocycles such as furan, thiophene and derivatives having at least one  $\alpha$ -free hydrogen are  $\alpha$ -mercurated very rapidly by the action of mercury (II) chloride and sodium acetate in the cold:

In an aqueous solution this method has been used for furan and 2-alkyl furan <sup>(8)</sup>. Also the halogenation

(replacement of mercury by halogen) <sup>(9)</sup> and symmetrisation <sup>(10)</sup> are a well known reactions of organomercury compounds through which aryl halogen and diaryl mercury are obtained respectively. **Experimental** 

#### 2-1 Materials & instruments

All chemicals and solvents have been supplied from FLUKA, BDH, ALDRICH companies . Infrared spectra have been recorded by Shemadzue FT-IR-8400S Fourier transform infrared spectrophotometers in the range between (4000-200) cm<sup>-1</sup> using KBr discs in chemistry department, college of scienceuniversity of Tikrit and UV spectra have been recorded also in the same department using UVvisible spectrophotometer Cintra 5GB scientific Equipment using DMSO and CHCl<sub>3</sub> as a solvents. CHN analyses have been carried out in chemistry AL-department. University of L- Baut-Jordan using instrument Euro vector EA 3000A Italy and the NMR spectra have been run in the same department by H-NMR Bruker Ultra shield 300 MHZ instrument using TMS as references and DMSO 6d as solvent and finally atomic absorption analysis has been done in research laboratories in the North gas - company -Kirkuk -Iraq.

## 2-2 Preparation of 2-Furyl $\alpha$ , $\beta$ -carbonyl compounds (1-4)<sup>(11)</sup>

Preparation of compound 1 in table 1 has been used as typical examples for the reaction: (3gm, 0.04 mole) of butanone and (4gm, 0.04 mole) of redistilled furfural have been introduced in small conical flask fitted with stoper, then 5ml of 10 % alcoholic KOH has been added then the mixture was stirred for 10 minutes at room temperature, then 10 ml of ethanol was added and the mixture was stirred for further 3hrs in an ice bath, the separated precipitate was filtered and washed with distilled water and cold ethanol, filtered, and dried, recrystallized from ethanol, weight recovered 4.78 gm, % yield 64%, M.P = (63- 65)C°.

## 2-3 Preparation of difurylidine and furylidine substituted benzylidines (5-9)<sup>(12)</sup>

Preparation of compound(6) Table (2) has been used as typical example (0.01mole,0.6gm) of acetone was introduced into a beaker (150ml) then 3ml of 25% alcoholic KOH was added and the mixture was stirred for 10 minutes then(0.01mole,1gm) of furfural and (1.4gm, 0.01 mole) of 4-dimethyl aminobenzaldehyde were added to the mixture followed by 10ml of ethanol, the mixture was stirred for 1hr at room temperature, then the precipitate was filtered and washed with water and cold ethanol, then dried in the oven at50C° then recrystallized from ethanol, weight recovered was 1.5gm, yield 56%.

## 2-4 Mercuration of the prepared compounds (1-9) using mercury(II) Chloride (10-19)<sup>(13)</sup>

Preparation of compound 10 in Table (3) has been used as typical example: (0.4gm, 0.002mole) of 1-(2-furyl)-1- pentene -3- one was dissolved in 5ml ethanol in a beaker(50ml), then (0.54gm, 0.002 mole) of mercury (II) chloride dissolved in 3ml distil. water was added followed by (0.16gm ,0.002mole) of sodium acetate dissolved in 3ml dist. water, then the mixture was stirred for 3hrs then left stand at room temperature for 24hrs. The precipitated product was filtered and washed extensively with hot water until the filtrate gave negative test for Hg<sup>++</sup> by aq. NaOH then the product was recrystallized from ethanol and weight recovered was 0.56 gm %yield 55% and M.P =89-91C°. One experiment has been done using benzal acetophenone (prepared according to reference <sup>(14)</sup> and the mercuration did not occure.

#### 2-5 Estimation of mercury by atomic absorption

100mg of the mercurated product was heated with 1ml of concentrated HCl and the same weight of the sample was treated with 7ml of distilled water (blank) and both samples were heated under reflux for 10hrs at  $80C^{\circ}$  in water bath then filtered in 100ml volumetric flask and completed at 100ml, then the concentration of Hg<sup>++</sup> was determined by atomic absorption instrument and the results are shown in table 2.

2-6 Preparation of symmetrized products (20-28)

The preparation of compound (20) Table (3) has been used as typical example for the reaction:

(0.14gm,0.001mole) of sodium iodide in acetone (10ml) was added to (0.4 gm,0.002mole) of compound (10) in acetone (15ml) in 50ml round bottomed flask and the mixture was stirred for 15 minutes and then left for 24hrs under stirring, the separated precipitate was filtered and washed with acetone and after recrystallization from ethanol and drying weight recovered was 0-32gm ,%yield 63%, M.P = 74-76C°.

## 2-7 Reaction of mercurated products with iodine (29-31)

The preparation of compounds (29) table (4) has been used as typical example for the reaction:

(0.178gm, 0.0007mole) of iodine in THF (5ml) was added to a small round bottomed flask containing (0.3gm, 0.0007mole) of mercurated product<sup>(10)</sup> in THF (10ml) then the mixture was stirred for 15min at room temperature and let stand for 24hrs. The precipitated product was filtered and washed with water then recrystallized from ethanol, dried, weight recovered was, % yield 62, M.P 81-83C°.

#### 2-8 Biochemical analysis

#### 2-8-1 Determination of Glucose in blood serum

After induction of diabetes mellitus in mice by alloxane the glucose level was determined in blood serum by enzymatic methods using kit of type (Randox, United Kingdom) through which the glucose is oxidized according to the following equation:

Ghucose + 
$$O_2$$
 +  $H_2O$   $\xrightarrow{glucose}$  gluconic acid +  $H_2O$   
2 $H_2O_2$  + 4-amino phenazone + phenol  $\xrightarrow{Peroxidas}$  ouinone imine +  $4H_2O$ 

#### Procedure

10  $\mu$ l from the blood serum and the standard and distilled water were taken separately in the clean and dry test tube and 1ml of RGT reagent was added as shown in table (5).

## 2-8-3 Determination Of triglyceride level in blood

The triglyceride level in blood serum was determined

using the kit from (Germany-Humane) using

enzymatic method according to the chemical

#### Procedure

10  $\mu$ l from the blood serum and the standard and distilled water were taken separately in the clean and dry test tube and 1ml of RGT reagent was added as shown in table (6).

$$\begin{array}{c} \text{Clycerol} + \text{ATP} & \xrightarrow{\text{Lipases}} & \text{Glycerol} + \text{Fatty acid} \\ \text{Glycerol} + \text{ATP} & \xrightarrow{\text{G.K.}} & \text{Glycerol} -3\text{-phosphate} + \text{ADP} \\ \text{Glycerol-3-phosphoate} + \text{O}_2 & \xrightarrow{\text{GPO}} & \text{dihydroxyacetone phosphate} + \text{H}_2\text{O}_2 \end{array}$$

### cholesterol + fatty acids

2-8-2 Determination of cholesterol in blood serum Cholesterol level was determined in the blood serum

by kit from (France -Biomaghrib) and also using

enzymatic methods according to the chemical

#### choles-4-en-one +H<sub>2</sub>O<sub>2</sub>

equation

serum

equations.

### 2H<sub>2</sub>O<sub>2</sub> + 4- aminoantipyrine POD

#### quinone imine + 4-chlorophenol

#### Procedure

10  $\mu$ l of the blood serum and the standard and distilled water were taken separately in the clean and dry test tube and 1ml of RGT reagent was added as shown in table (7).

#### **3- Results and Discussion**

## 3-1 Preparation of furyl $\alpha$ , $\beta$ -unsaturated compounds (1-9)

These compounds have been prepared according to

clasien-schmidt condensation using equimoler of aromatic or aliphatic ketone and aromatic aldehyde (furfural) in the presence of a base (10-25% KOH) using ethanol as solvent. The same method has been used to prepare diarylidine compounds using one mole of ketone (acetone or cyclohexanon) and two moles of similar or different aromatic aldehydes. The preparation of these compounds and the mechanism of the reaction are represented as shown in scheme 1.





Scheme 1: shows the preparation of furyl a, β-unsaturated compounds

The products have been identified by the IR spectra which showed strong absorption bands between 1591-1598 cm<sup>-1</sup> which belong to C=C olefinic double bonds and also strong absorption bands between 1643-1670cm<sup>-1</sup> due to the carbonyl groups conjugated with the double bond which are less than their normal absorption due to the conjugation<sup>(21)</sup>. The UV spectra showed bands in the range (227-293) nm ( $\pi$ - $\pi^*$ ) and bands in the range (297-368) nm (n- $\pi^*$ ) and showed red shift compared with unconjugated analogues. The I.R and U.V absorption are shown in table (8). The <sup>1</sup>H-NMR spectrum of compound (3) showed a doublet (1H) at 8.1 $\delta$  for C<sub>5</sub> and singlet (1H) for C4 at 6.7 $\delta$  of the furan ring and the protons of benzene ring and the olefinic protons showed absorption between  $7.4 - 7.9\delta$  for (6H).

#### 3-2 Mercuration of the compounds (1-9)

Mercuration of arenes is an electrophilic aromatic reaction and it proceeds for most aromatic compounds weather they as free benzenoid, poly aromatic or hetero aromatic species <sup>(14)</sup>, and according to that the compounds prepared in this research were mercurated due to the high susceptibility of furan ring to metalation by mercury salt:

$$\begin{array}{cccc} Fu\cdot R & + & HgX_3Fu\cdot R & & & H-Fu\cdot R & & & Fu-R & +H^*\\ & & & & & & & & \\ \pi-complex & & HgX & & HgX & \\ \end{array}$$

 $R = \alpha, \beta$ -unsaturated group ""

And the suggested mechanism could be represented as follow



As shown in the mechanism the mercury (II) chloride cation attacks the furan ring since the carbonyl is conjugated will carbon-carbon double bond so the carbonyl group tends to withdraw the electrons from the double and tends to deactivate it towards the electrophilic attack by HgCl<sup>+ (16)</sup>. All the mercurated products showed a significant I.R. absorption of – HgCl group in the range (349-310) cm<sup>-1</sup> (<sup>17)</sup> and the absorption of C-Hg band at (450-615) cm<sup>-1 (18)</sup> "also the spectra showed the carbonyl group absorption in the range (1712-1618) cm<sup>-1</sup> and also for the C=C double bond between (1658-1527)cm<sup>-1</sup> Table (9). The 1H-NMR spectrum of compound (13) showed two doublet at 7.6 and 7.9 $\delta(q)$  which belong to the olefinic double bond conjugated with the carbonyl group and proton H<sub>4</sub> and H<sub>3</sub> appeared at 6.8 and 7.38(d-d)(q) of furan ring respectively and the protons of benzene ring appeared at (8.28.3) $\delta$ (4H)(q), while compound (15) showed a sharp doublet at (2.92-2.98) $\delta$ (6H) (d) due to the two methyl groups attached to the nitrogen and 1,2 and 4,5 protons of olefinic double bond appeared between (7-7.5) $\delta$  and the protons 4<sup>-</sup> and 3<sup>-</sup> of furan ring appeared at 6.7 and 7 $\delta$ (q) while protons of benzene ring appeared between 6.5-7.7(4H)(m). Finally Table (10) showed CHN analysis of some of the prepared compounds. One experiment has tried using benzylidine acetophenone

and mercury II (chloride) using the same conditions for of furyl compounds, it was found that no mercuration was achieved according to the M.P, weight increase and IR of the product and the recovery of the starting material. Schemes 2,3.





R -- CHJCH3



Scheme 2: show the mercuration of the prepared compounds

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Scheme 3: shows the symmeteraisation of mercurated compounds

#### 3-3 Halogenation of mercurated compounds

The replacement of mercury by halogen takes place easily at room temperature due to the high ability for halogen to break C-Hg bond <sup>(19)</sup> and the IR spectra showed the disappearance of HgCl group absorption and the appearance of C-I absorption between (485-490)cm<sup>-1</sup>. The iodine analysis <sup>(20)</sup> for the iodinated compounds was shown in table (4).

#### **3-4 Symmetrisation reaction**

The symmetrisation products have been obtained by treatment of organomercury (II) Chlorides with NaI as a comlexing agent:

The products showed a significant decrease in the melting points indicating that the products  $(Het)_2Hg$  gained much more covalent character compared with the reactant (Het-HgCl), also the IR spectra showed the disappearance of Hg-Cl absorption, in addition to

the carbonyl absorption between (1618-1712)cm<sup>-1</sup> C<sup>----C</sup> and olefinic at (1525-1560) cm<sup>-1</sup> and absorption between 1475-1615cm<sup>-1</sup> due to C=C aromatic. The UV spectra also showed a higher  $\lambda$ max between (307-348)cm<sup>-1</sup>.

### 3-5 Biological activity

The  $\alpha,\beta$ -unsaturated compounds (3,4) have been tested for lowering the glucose, Cholesterol and triglyceride levels in blood serum of mice.

#### The effects of compounds 3 and 4

The present study showed that these compounds have significant effect in reducing glucose, total cholesterol and triglyceride levels in the serum test of laboratory mice. The reduction in glucose level was done after raising the glucose level in the blood of laboratory mice using alloxane material by injection process in the pretone. The results are shown in tables (11) and table (12).

Compd. No.	-R	Structure and name of product	Colour	m.p. °C	% Yield
1	-C <sub>2</sub> H <sub>5</sub>	CH <sub>2</sub> CH <sub>3</sub> 1-(2-furyl)-1-pentene-3-one	Dark red	65-63	63.7%
2	Br	(1-(p-bromophenyl)-3-(2-furanyl) -3-(2-furayl)2-propene-1-one	Golden yellow	83-81	62.6%
3		(1-(p-chlorophenyl)-3-(2-furanyl)- 3-(2-furayl)2-propene-1-one	Pale yellow	62-60	97%
4		(1-(p-nitrophenyl)-3-(2-furanyl)-3 -(2-furayl)2-propene-1-one	Dark brown	97-95	52%
5		1,5-di(furan-2-yl)penta-1,4-dien-3-one	Dark yellow	153-150	41.15%
6		CH <sub>3</sub> CH <sub>3</sub> 1-[p-(N,N-dimethylaminophenyl)-5- (furan-2-yl)penta-1,4-dien-3-one	Pale red	86-88	55.5%
7		1-(p-nitrophenyl)-5-(furan-2-yl)penta-1 ,4-dien-3-one	Pale orange	72-70	57%
8		1.(m-nitrophenyl)-5-(furan-2-yl)penta- 1,4-dien-3-one	Pale brown	130-129	98.3%
9		2-benzylidene-6-furylidene(cyclohexanone)	Dark brown	81-79	83.6%
	Ar =	),No <sub>2</sub> ,	, -	сн₃ сн₃	

Table 1	: Physical	properties of	f compounds	(1-9)

Fu = \_\_\_\_\_

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Table 2: P	hysical	properties of	Mercuration	products	(10-19)	)
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Compd No.	Reactant	HgCl <sub>2</sub> Wt(gm)/mol e	CH <sub>3</sub> COONaWt(g m) /mole	Structure and name of product	m.p °C	Yield %	Hg %
10	0.4/0.002	0.54/0.002	0.16/0.002	CI Hg 3-(5-chloro mercury-2-faryl)-1-ethyl- 2-p -2- propene-1-one	91- 89	55%	48 %
11	0.2/0.0007	0.18/0.0007	0.05/0.0007	CI Br 3-(5-chloro mercury-2-faryl)-1- bromophenyl)-2- propene-1-one	142 - 140	86.4 %	-
12	0.5/0.002	0.54/0.002	0.16/0.002	ci Hig Ci -ci -ci -ci -ci -ci -ci -ci -ci -ci -c	127 - 129	69%	32 %
13	0.5/0.002	0.54/0.002	0.16/0.002	CI Hg O NO <sub>2</sub> (5-chloro mercury-2-faryl)-1-(p- nitrophenyl)-2-propene-1-one	121 - 119	68%	39 %
14	0.4/0.002	0.5/0.002	0.16/0.002	cl Hg o chloro mercury-2-faryl)-1-(2- furyl)penta-1,4-dine-3-one	162 - 164	40%	
15	0.2/0.0007	0.18/0.0007	0.05/0.0007	CI	110 - 112	94.5 %	
16	0.7/0.002	0.54/0.002	0.16/0.002	CI Hg 3-(5-chloro mercury-2-faryl)-1-(p- nitrophenyl)-penta-1,4-dine-3-one	97- 99	69%	
17	0.7/0.002	0.54/0.002	0.16/0.002	3-(5-chloro mercury-2-faryl)-1-(m- nitrophenyl)-penta-1,4-dine-3-one	154 - 156 (d)	72%	
18	0.5/0.001	0.027/0.001	0.082/0.001	6-(5-chloro mercuryfurylidine-2- benzilidine cyclohexanone	93- 95	77%	
19	0.6/0.002	1.08/0.004	0.3/0.004	Bis-(5-chloromercury-2- farylidineacetone	273 - 275	47%	

R= -CH<sub>2</sub>CH<sub>3</sub>

Ar = \_\_\_\_ →Br · → -NO2

Table 3: Physica	l pro	perties of s	ymmeterisation	products	(20-28)
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Compd. No.	Reactant wt(gm)/mole	Sodumeioded Wt(gm)/mole	Structure and name of product	Colour	m.p. °C	Yield %
20	CI_Hg O CH <sub>2</sub> CH <sub>3</sub> 0.4/0.002	0.14/0.001	O CH <sub>2</sub> CH <sub>3</sub> ) <sub>2</sub> Hg Bis[5(1-ethyl)2-propene-1-one-3-yl)furan- 2-yl]mercury	red	74- 76	62.7%
21	сі вг 0.3/0.001	0.07/0.0005	Bis[5(p-bromophenyl)2-propene-1-one-3- yl)furan-2-yl]mercury	yellow	115- 113	77.5%
22	cicici 0.4/0.001	0.071/0.0007	chlorophenyl)2-propene-1-one-3-yl)furan- 2-yl]mercury	Pale yellow	93- 91	64%
23	сі по	0.11/0.0008	PNO2) Hg 2 Bis[5(1-(p- nitrophenyl)2-propene-1-one-3-yl)furan-2- yl]mercury	brown	108- 107	70%
24	сі ў Парадона 0.12/0.004	0.29/0.002	Bis[5-(5-furan-2-yl)penta-1,4dien-1-yl-3- one)furan-2-yl]mercury	yellow	141- 143	64%
25	сі 0.4/0.001	0.071/0.0005	Bis[5-(5-(p-N,N-dimethyl amino phenyl)penta-1,4dien-3-one-1-yl)furan-2- yl]mercury	brown	102- 103	70%
26	сі, на сі, сі, сі, сі, сі, сі, сі, сі,	0.14/0.0001	(5-(p-nitrophenyl)penta-1,4dien-3-one-1- yl)furan-2-yl]mercury	Pale red	85- 87	70%
27	ci_Hg 0 0.11/0.0002	0.14/0.0003	Bis[5-(5-(m-nitrophenyl)penta-1,4dien-3- one-1-yl)furan-2-yl]mercury	orange	153- 155	85%
28	сі, на община страна стран Страна страна стран Страна страна с	0.02/0.0002	Bis(5-(2-benzilidine)1,4—dine-3- cyclohexanone(furan-2-yl) mercury	Pale brown	95- 97	65.5%

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<b>Table 4: Physica</b>	I properties of iodinated	compounds (29-31)
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Compd. No.	Reactant wt(gm)/mole	I <sub>2</sub> Wt (gm)/mole	Structure and name of product	Colour	m.p. °C	Yield %	I %
29	сц <sub>на</sub> 0.3/0.0007	0.0007/0.17	3-(5-Iodo-2-furyl)-1-ethyl-2-propene-1-one	red	83- 81	62%	42%
30	сі ў сі на 0.2/0.0004	0.10/0.0004	3-(5-Iodo-2-furyl)-1-(p-chlorophenyl)-2-propene- 1-one	Brown	112- 110	44%	32%
31	CI_Hg 0.5/0.001	0.25/0.001	Bis[5(1-3-(5-Iodo-2-furyl)-1-(p-nitrophenyl)-2- propene-1-one	Dark brown	97- 95	92%	34%

#### **Table 5: Glucose determination**

	Standard	Sample	Reagent blank
Standard	10µl	-	-
Sample	-	10µl	-
Reagent 1	1000µl	1000µl	1000µl

#### Table 6: Cholesterol determination

	Blank	Standard	Sample
Standard	-	10µl	-
Sample	-	-	10µl
Working reagent	1ml	1ml	1ml

### **Table 7: Triglyceride determination**

	Blank	Sample	Standard
Sample	191	10µl	-
Standard	-	-	10µl
Reagent (RGT)	1000µl	1000µl	1000µl

### Table 8: Spectral properties of products (1-9)

Compd.		$U_{\rm V} \lambda (\rm max) \rm nm CHCh$			I.R(KBr), v(cr	n <sup>-1</sup> )
No.	Structure and name of product	π*-π , n- π*	υC=Ο	υC=C	$_{vAr}C = C$	Others
1	CH <sub>2</sub> CH <sub>3</sub>	227,297	1643	1595	1552	υCH <sub>2</sub> CH <sub>3</sub> 2847
2	Contraction of the second seco	298,305	1670	1593	1479	C-Br 735
3		291,300	1654	1598	1556	C-Cl 714
4		279,302	1658	1591	1483	-NO <sub>2</sub> sym , asy 1344,1523
5		225,368	1698	1662	1554	-
6		272,300	1687	1652	1600	1,4-disah 825
7		280,306	1660	1591	1471	-NO <sub>2</sub> sym , asy 1328,1500
8	NO2	284,301	1690	1658	1618	-NO <sub>2</sub> sym , asy 1348,1500
9		293,313	1656	1612	1560	υCH <sub>2</sub> 2811

Compd.		U.v. λ(max)nm CHCl <sub>3</sub>	I.R(KBr), υ(cm <sup>-1</sup> )		
No.	Structure and name of product	π*-π , n- π*	υC=Ο	υC=C	vHg-Cl
10	CI Hg O CH2CH3	297,307	1706	1616	327
11	CI_Hg O Br	292-328	1670	1595	310
12	CI Hg O CI	296-348	1658	1596	327
13		286-301	1660	1591	349
14	CI I I I I I I I I I I I I I I I I I I	267-290	1712	1608	317
15		324-364	1672	1658	345
16		275-298	1664	1595	335
17		290-312	1618	1527	328
18		286-316	1654	1608	319
19		283,305	1712	1606	340

	<b>Table 9: Spectral</b>	properties of	products	(10-19)
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### Table 10: Elemental analyses of compounds (3,4,12,13)

Compd.	Calculation (Exp.)		Theoretical			
No.	C%	H%	N%	C%	H%	N%
3	67.24	3.85	00.0	67.14	3.87	0.00
4	64.22	3.70	5.74	64.20	3.70	5.76
12	33.43	1.76	0.00	33.40	1.71	0.00
13	32.53	1.81	2.88	32.67	1.68	2.93

Table 11:	Biologica	l activity o	f com	pound	3
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	Triglycerides	Total cholesterol	Glucose
	mmol/liter	mmol/liter	mmol/liter
Control group	39.00±0.84	132.55±7.99	112.46±1.75
Control group	· b	В	b
Affected mice without	43.90±0.56	187.60±3.25	424.80±53.17
treatment	a	a	а
Tracted offected miss	36.11±1.57	128.93±7.33	95.90±14.99
Treated affected mice	с	b	В

Tuble 12. Diological activity of compound 4					
	Triglycerides	Total cholesterol	Glucose		
	mmol/liter	mmol/liter	mmol/liter		
Control group	39.00±0.84	132.55±7.99	112.48±1.77		
Control group	b	b	b		
Affected mice without	43.9±0.56	187.60±3.25	424.80±53.17		
treatment	а	a	а		
Transferd offerstad miles	27.05±0.49	138.70±3.96	123.30±8.34		
I reated affected mice	с	b	b		

Table 12: Biological activity of compound 4

#### References

1) A. J. AL-Hamdany, ph. D. thesis, university Mosul, Mosul, Iraq, p34(2002).

2) D. N. Dhar "the chemistry of chalcones and related compounds", John wiley and sous, Inc., 214-215(1981).

**3)** L. Zhai, M. chem, T. Blon. G. thender S. D. Christensen and A. Kharazni, journal of antimicrobial vhemotherapy, 43, p793-803,(1999).

4) H. Nagaraj and C. S. Reddy, J. Iran. Chem. Soc., vol., No.2, p262-267(2008).

5) K. Kamor. A. Kumar and G. praiapat "An efficient conjugate addition of an activated nucleophilic carbamate with chalcones" p.179 (2004).

6) H. Makita, cancer Res. 56, p.4904-4909(1996).

7) O. V. Yarishkin, Bio. Org. Med. Chem. Lett., 18, P.137-140(2008).

8) H. Gilman and G. F. Wright, J. Amer. Chem. Soc., 55,3302,1938.

9) F. R. Jensen and B. Rickbone "Electrophilic substitution of organomercuiral, Me Graw. Hill, New York (1968).

10) F. C. Whitmore and R. J. Sobatzi, J. Am. Chem. Soc. 55,1128(1933).

11) N. L. Shihabaddin, ph. D. thesis, Basrah university, Iraq, (1998).

**12)** C. S. Marrel, L. C. Coleman, J. R. and G. P. Scott, J. Organic Chemistry, 20,1785-1792(1992).

13) L. C. Makarova and A. N. Nesmeyanove, "the organic compounds of mercury" pub-North-Holland, Amsterdam, p.118(1967).

14) A. I. Vogel "Practical organic chemistry" Longmans, green and Co.718(1956).

15) Comprehensive organometallic chemistry volume 2, P. 874,1982.

16) D. N. Dhar "the chemistry of chalcones and related compounds", John wiley and sous, Inc., p.54-57(1981).

17) A. AL-Kadhimi, ph. D. thesis, university of Lancaster, UK, p.133(1985).

18) N. A. AL-Jabar, ph. D. thesis, university of Technology, UK, p.102(1985).

19) R. C. Larock, Tetrahedron, 38,1715(1982).

20)G. Skoog, W. Ward "Fundamental of analytical chemistry" 6<sup>th</sup> ed. Arnold.

**21)**D. H. Williams and I. Fleming "Spectroscopic methods in organic chemistry" 4<sup>th</sup> ed., Mc Graw. Hill, maidenhead, 315(1987).

### تحضير ، زئبقة وهلجنة لبعض من كيتونات الفيوران غير المشبعة الفا ، بيتا ودراسة الفعالية الداملوحية لها

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#### الملخص

نتاول الجزء اللاول من البحث تحضير بعض مركبات β،α-كاربونيل غير المشعة الحاوية على حلقة الفيوران في وسط قاعدي المركبات (1-4) وايضاً بعض مركبات الثنائي فيوريلدين والفيوريلدين-بنزلدين و التي تم تحضيرها من الفورفورال والالدهيدات معوضة اضافة الى السايكلوهكسانون المركبات (5-9) في وسط قاعدي أيضا. أما الجزء الثاني من البحث فقد تضمن زئبقة المركبات المحضرة باستخدام كلوريدالزئبقيك المركبات (01-(10) والتي اعقبها تفاعل تماثل (Symmeterisation) باستخدام يوديد الصوديوم للحصول على مركبات الزئبق المتماثلة (Fur-Hg-Fur) أو (Fu-Hg-Ph) وتم استبدال مجاميع الزئبق المعوضة في بعض المركبات بمجاميع اليود بالتفاعل مع اليود المركبات (20-الثالث من البحث فقد تتاول اختيار الفعاليه البيولوجيه للمركبات (3-9). تم تشخيص المركبات المحضرة باستخدام تفنية R تقنيات HNMR,CHN,UV.