SYNTHESIS AND CHARACTERIZATION OF SOME NEW BIS-CHALCONESAND THEIR TRANSFORMATION TO BIS-THIOCARBAMOYL-2- PYRAZOLINE DERIVATIVES

Farouq **E**mam **H**awaiz[1], **F**aiq **H**amasaed **H**ussein [2] and **H**ashim **J**alal **A**zeez[1]
[1]Chemistry Dept. College of Education, University of Salahadin-Hawler, Erbil -Kurdistan -Iraq
[2]Chemistry Dept. College of Science, University of Salahadin- Hawler, Erbil -Kurdistan -Iraq
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Abstract

The starting materials alkoxy and benzyloxyacetophenones (1a-j, 2 and 3) have been prepared on the basis of Williamson synthesis of ether. The prepared and the available starting materials were reacted with terphthalaldehyde using green methods, Ultrasound , Microwave and solvent-free grinding method to give anew series of bis- chalcones (4a-s).The prepared bis- chalcones were subjected to react with thiosemicarbazide according to the Michael addition reaction to afford new biologically active bis-pyrazolinedeivatives(5a-s). The structure of the synthesized compounds were characterized by spectral methods: FT-IR, ^{1}H-nmr, ^{13}C-nmr andMass spectrometry.

Keywords*: Microwave, Ultrasound,Grinding,Chalcone, Thiosemicarbazide, Pyrazoline.*

Introduction

Chalconesare open chains C_6-C_3-C_6(1, 3-diaryl-2-propen-1-ones) constitute a class of naturally occurring and synthetic compounds, the first intermediates of flavonoid biosynthesis. They occur sporadically in plants as yellow pigments usually give yellow to orange color to the tissues in which they are located[Buckingham 1994]. Chalcones can easily be synthesized on the bases of Claisen-Schmidt condensation reaction[Mirjalil and Zaghaghi 2008] and can be used as a precursor for the preparation of different important heterocyclic compounds like; flavonoid[Sayed 2004],[Soon et al] and pyrazolines[Azarifarand Shaebanzadeh 2002],[Goda et al]. Pyrazolines are the five membered heterocyclic compounds containingtwo adjacent nitrogen atoms[Otera (2000], most commonly prepared from chalcones according to the Michael addition reaction[Li et al1995].

Both chalcones and pyrazolines possess a wide spectrum of biological activity which include : antifungal [Charles et al 1987],[Ozdemir et al 2010] anti-inflammatory[Hsieh et al 1998],[Sahu et al 2008] ,analgesic[Viana et al 2003],[Abd 2008], antileshmanial[Nielsen et al 1998],[Mei 2003], antioxidants[Miranda et al 2000],[Azizur et al 2010],and antimicrobial activities[Biswajit et al 2010],[Nada et al 2008]. Accordingly, herein we have described different methods for the preparation of some new bis-chalcones and their transformation to pyrazoline derivatives.

Experimental

Melting points were determined using an Electrothermal melting point apparatus, IR spectra were recorded on a Bio-rad Merlin FT-IR spectroscopy Mod FTS 3000, using KBr disc. ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker(300MHz) with TMS as internal reference. Mass Spectra (MS) were recorded on GCMS, Shimadzu, QP 5050A and LC/MS/ API 5000.The Sonication was performed by Telsonic Ultrasonic type Tpc-25-2. A6 CH-9552 Bronschhofen, out put 75/150w , 30 KHz. The microwave irradiation was carried out by domestic microwave oven PM-001W pacific, 700 w, 2450MHz.

1. Preparation of starting materials:

1.1 Preparation of 4-alkoxyacetophenones[Vanstone et al 1989] (1a-j) :

A mixture of 4-hydroxyacetophenone (1.36 gm , 0.01 mol), an appropriate alkyl bromide (0.012 mol) and anhydrous potassium carbonate (4.2gm , 0.03 mol)in acetone (30ml) was refluxed with stirring, progress of the reaction being monitored by TLC for disappearance of starting materials. The cooled mixture was poured into water and extracted by ethylacetate (2 x 50ml).The combined organic extracts were washed with water , dried over Na_2SO_4, the mixture then filtrate and the solvent was evaporated in rotary evaporator .The residue was

dissolved in ether and purified by flash chromatography to give the pure 4-alkoxyacetophenones (1a-i),the melting points between (28-30 and 35-37^{o}C). , yields (73% to 87%), reaction times (3 to 5hrs.) and IR data C=O: 1676, C=C: 1601cm^{-1}.

1.2 Preparation of 4-benzyloxy and 4-(4-chlorobenzyloxy) acetophenone (2 and 3):

According to the modified procedures[21] a solution of 4- hydroxylacetophenone (1.36gm , 0.01mol), alkyl halide (0.015mol)and anhydrous K_2CO_3 (4.2gm , 0.03mol) in absolute ethanol (20ml) was refluxed with stirring for six hours. When all starting materials had reacted which was monitored by TLC.The cooled mixture was poured into water, solid materials immediately was obtained. The desired products was filtered off , washed several times with water and cold ethanol, dried and recrystallized from ethanol (charcoal) to obtain white crystals of compound (2): m.p. (90-92 ^{o}C), yield (2gm, 88.3 %).IR(cm^{-1}); 1671 (C=O), 1598 (C=C), 1263 and 1180 (C-O-C) stretching, and compound (3) m.p. (85-87 ^{o}C), yield (2.5gm, 96 %), IR(cm^{-1}) ; 1667 (C=O), 1597 (C=C), 1263 and 1175 (C-O-C) stretching.

2. Synthesis of Chalcones:

2.1 Synthesis of 1,4-bis [3-oxo-3(4-substitutedphenyl)propenyl] benzene (4a-s):

2.1 Method (A):Ultrasound - assisted synthesis of bis-chalcones[Guofeng et al 2004]:

Terephthalaldehyde (0.134gm ,0.001mol)was dissolved in ethanol (5ml)and added to the solution of (0.002mol) of an appropriate substituted acetophenones in ethanol (5ml) and 2ml of (4% ethanolic NaOH dissolved using ultrasound).The mixture was irradiated by ultrasonic bath at room temperature for (30s. to 5 min.).The mixture was solidified and the pale yellow chalcones were separated by suction filtration , washed with ethanol and water to neutralize ,dried and purified by recrystallization from a suitable solvent (toluene or ethanol). The results were tabulated in Table (1).

2.1 Method (B): Solvent-free trituration synthesis of bis-chalcones[Gareth & Colin 2001](4a-n):

Terephthalaldehyde (0.134gm, 0.001mol) , liquid substituted acetophenones (0.002mol) and solid NaOH (0.08gm, 0.002mol) were combined using a mortar and pestle; the pale yellow medium was converted to powder product through (5 to10min.),the products were washed with water to neutralize and recrystallized from toluene.

2.1 Method (C):Microwave –assisted synthesis of bis-chalcones (4o-s):

Terephthalaldehyde (0.134gm, 0.001mol) , solid substituted acetophenones (0.002mol) and 2gm of K_2CO_3 were combined using a mortar and pestle. The mixture was transferred to a (50ml) beaker, and placed vertically in the center of the microwave oven and irradiated at (390watt) for (1-4)successive periods of 30s with mixing by glass rod between the successive periods until the reaction was completed as a yellow powder.

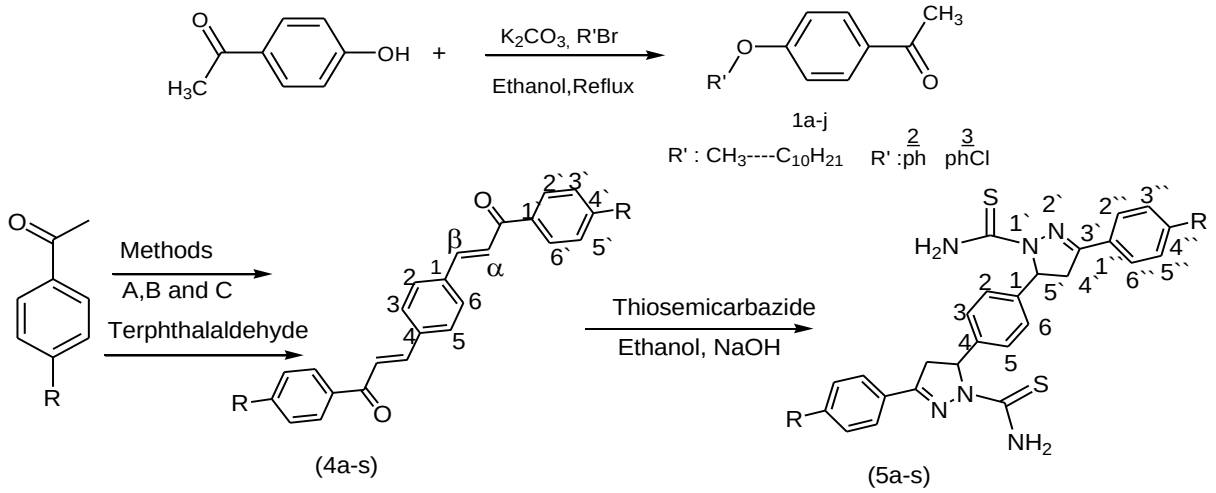
3. Synthesis of Thiocarbamoyl-2- Pyrazolines

Synthesis of 1,4-bis[3-(4-substitutedphenyl)-1-thiocarbamoyl-2-pyrazolin-5-yl] Benzene[Palaska et al 2003](5a-s)

A mixture of bis-chalcone (0.0005mol), thiosemicarbazide(0.091gm, 0.001mol) and sodium hydroxide (0.04gm, 0.001mol) in ethanol (15ml) was refluxed with stirring for an appropriate time. The mixture was cooled, the resulting precipitate isolated by suction filtration, washed with water to neutralize and then with ethanol. The product was dried and recrystallized from ethanol or washing with hot ethanol. The time, m.p. and the percentage of yields are summarized in Table (3).

Results and Discussion

In this study we utilized three different methods like solvent-free, ultrasound, and microwave irradiation techniques to obtain a series of new bis- chalcones from the reaction of terphlaldehyde and different substituted acetophenones containing either electron - releasing or electron- withdrawing groups, to provide the products in excellent yields and short reaction times and in order to draw a qualitative and quantitative comparison between the methods together and with previous reports. The results showed that the green methods are fast, clean and efficient, relatively high yields were achieved in short reaction times. The prepared bis- chalacones were converted to bis-thiocarbamoylpyrazolines by reacting with thiosemicarbazide as shown in Scheme (1).



R: a OCH3, b OC2H5, c OC3H7, d OC4H9, e OC5H11, f OC6H13, g OC7H15, h OC8H17, i OC9H19, j OC10H21.
k H, l CH3, m F, n Cl, o Br, p 3,4(OMe)2, q 2-Naph., r BzO, s ClBzO.

Scheme (1)

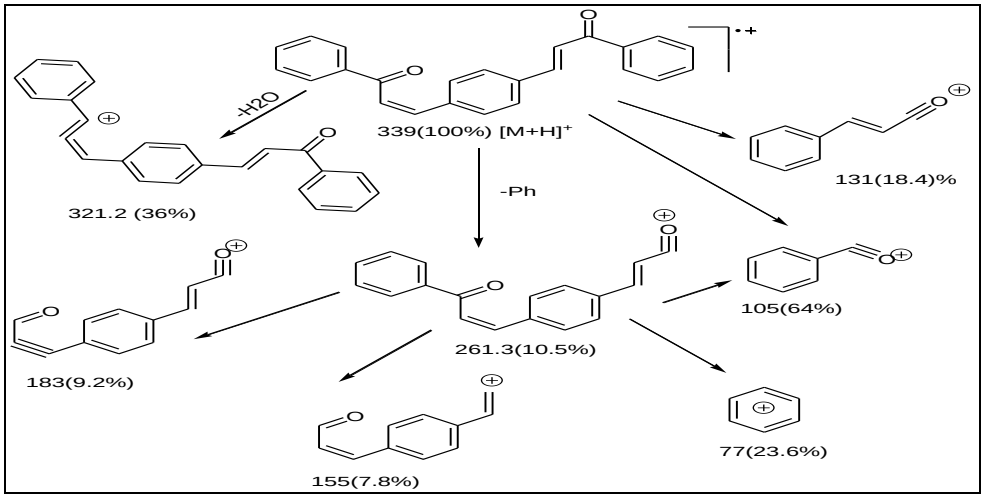
The comparison between the three efficient methods together and with classical methods has also been studied in terms of reaction times and product yields .It was found that in the presence of solvent (ethanol) all reactants were succeeded with ultrasound irradiation method in 0.5—5 minutes while in the case of solvent-free conditions method- (B) grinding by mortar and pestle succeeded and gave excellent yields only when both reactants are liquids or at least one of them is liquid. Moreover, such reactions and conversions were carried out successfully with-in 1-3 minutes in very good to excellent yields under solvent-free microwave irradiation. Thus indicate that the effect of microwave irradiation is not purely thermal. Microwave irradiation facilitates the polarization of the molecules under irradiation causing rapid reaction to occur[Katritzky2005]. All physical data are summarized in Table (1) .The formation of the intermediates bis-chalcones (4a-s) and bis-pyrazolines (5a-s) were confirmed on the basis of their spectral data IR , ^{1}H-NMR , ^{13}C-NMR ,and mass spectra . In the IR spectra of bis-chalcones,Table (3), the shifting of the absorption band of carbonyl group of the two reactants, substituted acetophenones from 1676-1679 cm^{-1} and terphthalaldehyde to lower wave numbers 1662-1653cm^{-1}; this is a strong evidence for the formation of conjugated enone of chalcones[Mao&George 2000]. Other strong band at 1597-1607 cm^{-1} corresponding to carbon-carbon double bond of enone and aromatic rings, and two strong bands for alkoxy group of chalcones appeared at 2878- 2853 cm^{-1} and 2965-2922 cm-1. The IR spectra of bis-thiocarbamoylpyrazolinesTable(3), were very informative and furnished good evidence for the formation of expected structures , all spectra shows three characteristic bands at (3391-3488cm^{-1}), corresponding to the NH_2 stretching vibrations of thiocarbamoyl group attached to pyrazoline ring[Stuart 2004]. A second two important bands at 1565- 1607 and 1466-1511 cm^{-1} attributed to C=C and C=N. Also, all compounds show intense band in the region 1346- 1374cm^{-1} belonging to the C=S stretch of thiocarbamoyl group[Asha&Amir2006]. The disappearance of characteristic carbonyl group band of conjugated system and the appearance of imine band are also good evidence for confirming the pyrazoline structure. The ^{1}H-NMR spectra of chalcones, Table (4),Figure(1) showed that the Cα-H and Cβ-H protons are considerably shifted downfield to the extent that they appear in aromatic region δ 6.6-8.0

As a result, these protons can hardly be distinguished from those of the aromatic ring protons. This is probably associated with the joint deshielding resonance and anisotropic effects of the phenyl ring bonded to β- carbon atom [Azarifar&Shaabanzadeh 2002]. Also the disappearance of aldehydic protons of the reactants at 9-10 indicated the formation of desired products. The most important features of ^{13}C-NMR spectra of bis-chalcones Table (5),

Figure (2) are the C-β carbon atom resonance at δ (139.64—143.56) appeared downfield of those of the C-α atoms at (120.67—123.52ppm) because of the mesomeric de-shielding effect of the carbonyl group. Further support for the structural elucidation comes from mass spectra of some compounds. The Mass spectra and of some chalcones Figure (3), were observed excellent agreement between found and expected spectra. Thus it is considered as another confirmation for the structural elucidation of the prepared chalcones, Scheme (2). In the ^{1}H-NMR spectra Table (4), Figures (4) the (ABX) spin system of pyrazoline ring was observed. The pyrazoline protons H_A and H_B are geminal protons at C-4` carbon appear in the region of δ (3- 3.15) and δ (3.76- 3.9) as doublet of doublets. The C-H protons of C-5` also appears as doublet of doublet (dd) in the region of δ (5.8-6) attributed to the vicinal coupling with two nonequivalent geminal protons of C-4` carbon .Also all compounds appeared a doublet broad band at δ (7.84- 8.2) corresponding to the NH_2 protons of thiocarbamoyl group and a characteristic bands for each of substituents were appeared .

^{13}C-NMR chemical shift values of the carbon atom at δ 42.78- 42.89 for C-4`, δ 62.63- 63.22 for C-5` and δ 154.48- 155.58 for C-3` corroborate the 2-pyrazolines character are deduced from the ^{1}H-NMR data and a characteristic band at δ 176.4- 178.37 corresponding to C=S carbon resonance[Seebacher et al 2003] confirms the structure of thiocarbamoyl-2-pyrazolineTable(5).

The mass spectra of pyrazolines represented by compound 125k Figure (5) shows a molecular ion peak at m/z 485.2 (56.6%) [M+H] confirming its molecular weights. The fragmentation started by initial loss of the NH_2 group to give molecular ion at m/z 468(100%) as a base peak and was followed by loss of phenyl ring to obtain m/z 392.3 (23.3%) . The second thiocarbamide group was lost and followed by other fragmentations or the fragmentation may be started by initial loss of SH group[Asha& Amir 2007] obtained an ion at m/z 450.9(44.7%) and the loss of (CN) group to give m/z 426.6 (8.3%) and losses of a second (NH_2) group gave m/z 409.3 (26.6%) and followed as above .



Scheme (2): Mass fragmentation of chalcone (4k)

Table (1): Some physical properties of the synthesized bis- chalcones (4a-s):

Prod.(4)	R	Molecular formula	M.P. °C	Yield %		
				A	B	C
A	OCH_3	$C_{26}H_{22}O_4$	244-246	95.4	92.9	
B	OC_2H_5	$C_{28}H_{26}O_4$	210-212	86.5	85.6	
C	OC_3H_7	$C_{30}H_{30}O_4$	198-199	88.1	90.3	
D	OC_4H_9	$C_{32}H_{34}O_4$	196-198	85.0	82.9	

E	OC_5H_{11}	$C_{34}H_{38}O_4$	186-187	86.2	86.2	
F	OC_6H_{13}	$C_{36}H_{42}O_4$	182-184	89.2	87.3	
G	OC_7H_{15}	$C_{38}H_{46}O_4$	185-187	84.8	83.0	
H	OC_8H_{17}	$C_{40}H_{50}O_4$	172-174	87.5	85.8	
I	OC_9H_{19}	$C_{42}H_{54}O_4$	167-168	86.8	86.0	
J	$OC_{10}H_{21}$	$C_{44}H_{58}O_4$	160-162	86.1	85.3	
K	H	$C_{24}H_{18}O_2$	196 -197	97.6	94.6	
L	CH_3	$C_{26}H_{22}O_2$	228-230	90.3	92.8	-------
M	F	$C_{24}H_{16}F_2O_2$	238-240	90.9	92.2	-------
N	Cl	$C_{24}H_{14}Cl_2O_2$	248-250	88.4	86.0	
O	Br	$C_{24}H_{14}Br_2O_2$	276-278	84.6	-------	82.6
P	3,4-diOCH_3	$C_{28}H_{26}O_6$	220-222	85.1	-------	84.0
Q	2-Naph.	$C_{32}H_{22}O_2$	224-226	89.0	-------	89.0
R	BzO	$C_{38}H_{30}O_4$	210-212	89.0	-------	87.2
S	4-ClBzO	$C_{38}H_{28}Cl_2O_4$	200-202	87.2	-------	88.8

Table (2): Some physical properties of the synthesized bis-thiocarbamoyl pyrazolines (5a-s)

Prod.(5)	OR	Molecular formula	M.P./dec. °C	Time (hrs.)	Yield %
A	CH_3	$C_{28}H_{28}N_6O_2S_2$	228-230	4.5	73.5
B	C_2H_5	$C_{30}H_{32}N_6O_2S_2$	264-266	5.0	73.4
C	C_3H_7	$C_{32}H_{36}N_6O_2S_2$	245-247	6.0	70.0
D	C_4H_9	$C_{34}H_{40}N_6O_2S_2$	240-242	6.0	73.2
E	C_5H_{11}	$C_{36}H_{44}N_6O_2S_2$	240-241	6.0	70.0
F	C_6H_{13}	$C_{38}H_{48}N_6O_2S_2$	242-244	6.5	67.2
G	C_7H_{15}	$C_{40}H_{52}N_6O_2S_2$	238-240	6.5	61.7
H	C_8H_{17}	$C_{42}H_{56}N_6O_2S_2$	240 dec.	7.0	62.0
I	C_9H_{19}	$C_{44}H_{60}N_6O_2S_2$	230 dec.	7.0	65.0
J	$C_{10}H_{21}$	$C_{46}H_{64}N_6O_2S_2$	150-152	7.0	65.2
K	H	$C_{26}H_{24}N_6S_2$	230dec.	4.0	86.7
L	CH_3	$C_{28}H_{28}N_6S_2$	280	5.0	85.9
M	F	$C_{26}H_{22}F_2N_6S_2$	278	5.0	88.4
N	Cl	$C_{26}H_{22}Cl_2N_6S_2$	274	5.5	81.3
O	Br	$C_{26}H_{22}Br_2N_6S_2$	260	6.0	74.7
P	3,4-di-OCH_3	$C_{30}H_{32}N_6O_4S_2$	264	6.0	72.8
Q	2-Naph.	$C_{34}H_{36}N_6S_2$	258	6.5	75.3
R	BzO	$C_{40}H_{36}N_6O_2S_2$	218	7.5	71.8
S	4-ClBzO	$C_{40}H_{34}Cl_2N_6O_2S_2$	210	8.0	70.5

Table (3): Assignment of characteristic frequencies (cm^{-1}) of IR spectra for the prepared chalcones (4a-s) and pyrazolines (5a-s):

Prod.	4a-j			5a-j		Prod.	4k-s			5k-s	
	C=O	**C=C**	**N-H**	**C=N**	**C=C**		**C=O**	**C=C**	**N-H**	**C=N**	**C=C**
a	1654	1599	3302	1606	1515	k	1655	1606	3349	1608	1513
b	1654	1604	3352	1607	1514	l	1655	1598	3348	1607	1515
c	1657	1603	3350	1607	1514	m	1662	1603	3348	1603	1511
d	1655	1603	3350	1602	1514	n	1656	1606	3344	1609	1513
e	1657	1603	3350	1604	1515	o	1655	1607	3346	1609	1513
f	1657	1603	3351	1607	1515	p	1654	1597	3352	1607	1513
g	1653	1603	3351	1605	1515	q	1658	1606	3339	1606	q
h	1657	1603	1652	1607	1514	r	1657	1602	3339	1607	1514
i	1658	1604	1651	1605	1514	s	1653	1600	3335	1607	1514
j	1655	1603	1552	1604	1513	---	------	-----	----	----	-----

Table (4): The ^{1}H-NMR data for the prepared bis-chalcones(4b, k,l,p) and bis-pyrazolines (5I, k,l):Solvent $CDCl_3$.

Prod.	δ ppm Multiplicity Intensity Assignment
4b	1.07 (t, 6H, 2 CH3 –C9); 1.87 (sextet, 4H, 2 CH2 –C8); 4.03 (t, 6H, 2 CH2 –C7); 6.99 (d, 2H, 2 H-α); 8.06 (d, 2H, 2 H-β); 7.2- 7.85 (m, 12H, Ar-H)
4k	7.55 (d, 2H, 2 H-α); 7.95 (d, 2H, 2 H-β); 7.4-7.9 (m, 14H, Ar-H)
4l	2.47 (s, 6H, 2 CH3- C4`) ; 7.34 (d, 2H, 2H-α) ,7.97 (d, 2H, 2 H-β); 7.2- 7.84 (m, 12H, Ar-H).
4p	3.96 (s, 12H, 4 OCH3 –C3`,C4`); 6.95 (d, 2H, 2 H-α), 7.73 (d, 2H, 2 H-β); 7.28- 7.85 (m, 10H, Ar-H)
5i	3.13(dd, 2H, 2 HA) ; 3.8 (s, 6H, 2OCH3-C3``,C4``) ;3.80(dd, 2H, 2HB) ; 5.90(dd, 2H, 2 Hx); 6.96- 8.08(m, 12H, Ar-H``) ; 8.21 (b, 4H, 2NH2).
5k	3.13(dd, 2H, 2 H_A) ; 3.90(dd, 2H, 2H_B) ; 5.92(dd, 2H, 2 H_x); 7.09- 7.94 (m, 14H, Ar-H) 8.16, 8.2 (bb, 4H , 2NH_2).
5l	2.33(s, 6H, 2CH_3-4`` ) ; 3.1(dd, 2H, 2 $H_A$) ; 3.84(dd, 2H, 2$H_B$) ; 5.89 (dd, 2H, 2 $H_x$); 7.07(s, 4H, 2 Ar-$H_{2,3,5,6}$); 7.24 (d, 4H, 2xAr-$H_{3``,5``}$); 7.75 (d, 4H, 2Ar-$H_{2``,6``}$); 7.84, 7.98 (bb, 4H, 2$NH_2$).

Table (5): The^{13}C-NMRdata for the prepared bis-chalcones(4k,l,p) and bis-pyrazolines (5 l,m) ; Solvent $CDCl_3$.

4k		4l		4p		5l		5m	
δ	**Assig.**	**δ**	**Assig.**	**δ**	**Assig.**	**δ**	**Assig.**	**Δ**	**Assig**
123.5	C-α	21.71	C_7	56.1	C_7	21.52	CH_3	42.78	$C_{4`}$
129.0	$C_{2,3,5,6}$	123.0	C-α	109.9	$C_{3`}$	42.78	$C_{4`}$	55.85	OCH_3
129.2	$C_{3`,5`}$	128.6	$C_{2,3,5,6}$	110.7	$C_{6`}$	62.89	$C_{5`}$	63.09	$C_{5`}$
129.9	$C_{2`,6`}$	128.9	$C_{2`,6`}$	122.5	$C_{2`}$	125.99	$C_{2,3,5,6}$	114.28	$C_{3``,5``}$
133.7	$C_{1,4}$	129.9	$C_{3`,5`}$	123.1	C-α	127.59	$C_{1``}$	123.75	$C_{1``}$
137.1	$C_{4`}$	135.5	$C_{1`}$	128.8	$C_{2,3,5,6}$	128.56	$C_{2``,6``}$	126.17	$C_{2,3,5,6}$

137.9	$C_{1`}$	136.9	$C_{1,4}$	131.1	$C_{1`}$	129.71	$C_{3``,5``}$	131.49	$C_{2``,6``}$
143.5	C-β	143.1	C-β	136.9	$C_{1,4}$	140.95	$C_{4``}$	142.38	$C_{1,4}$
189.6	C=O	143.1	$C_{4`}$	142.7	C-β	142.03	$C_{1,4}$	155.31	$C_{3`}$
		189.7	C=O	149.3	$C_{5`}$	155.58	$C_{3`}$	161.66	$C_{4``}$
				153.4	$C_{4`}$	176.40	C=S	178.37	C=S
				188.3	C=O				

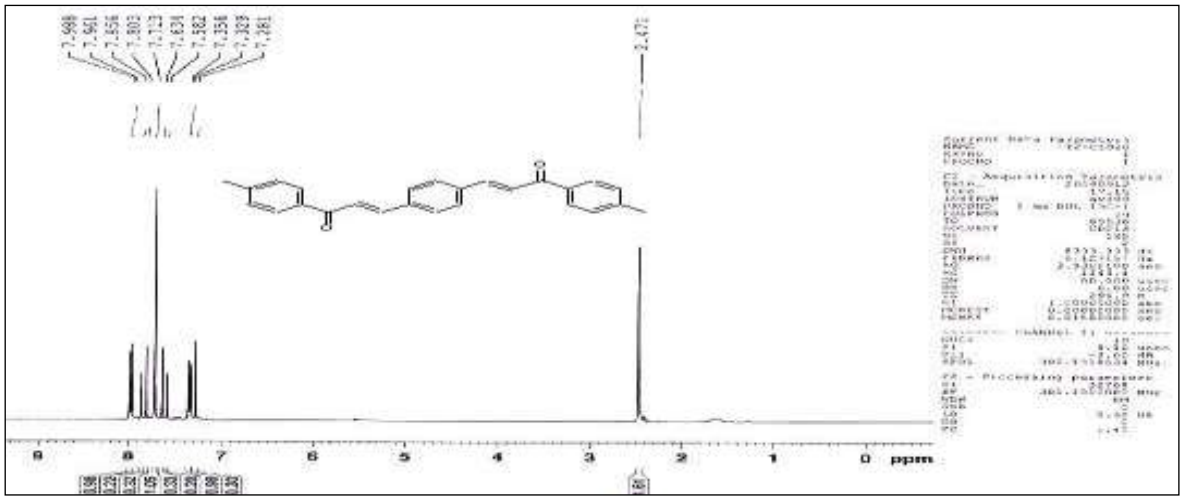


Figure (1): ^{1}H-NMR spectrum of compound (4l)

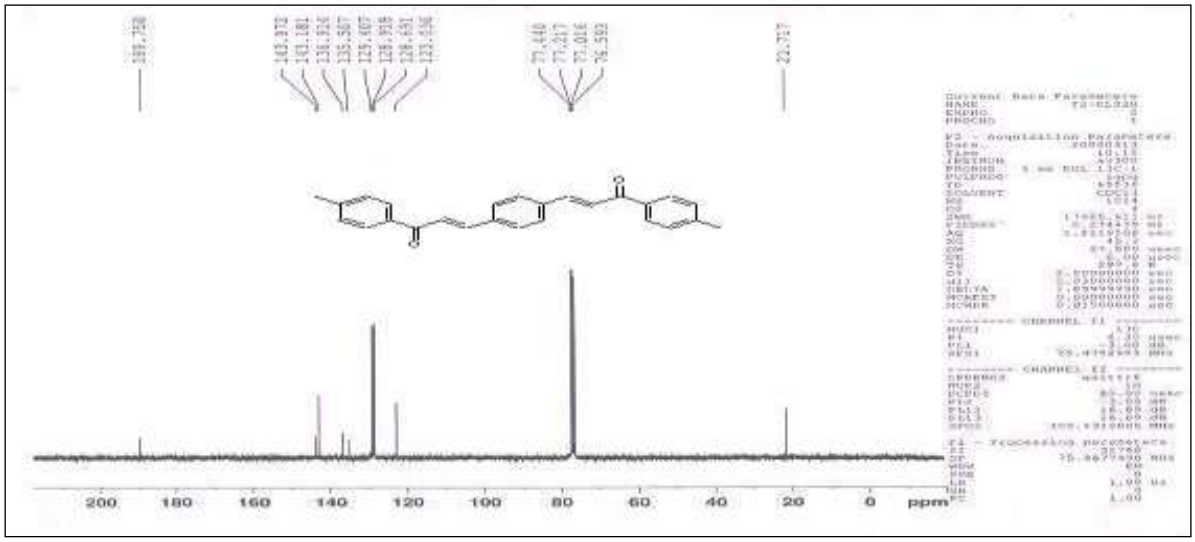


Figure (2): ^{13}C-NMR spectrum of compound (4l)

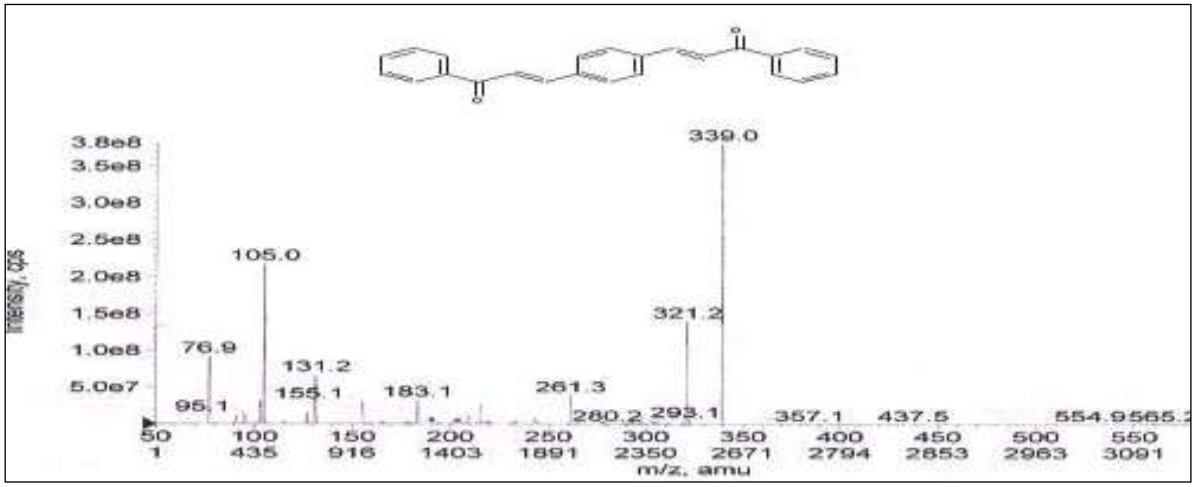


Figure (3): Mass spectrum of compound (4k)

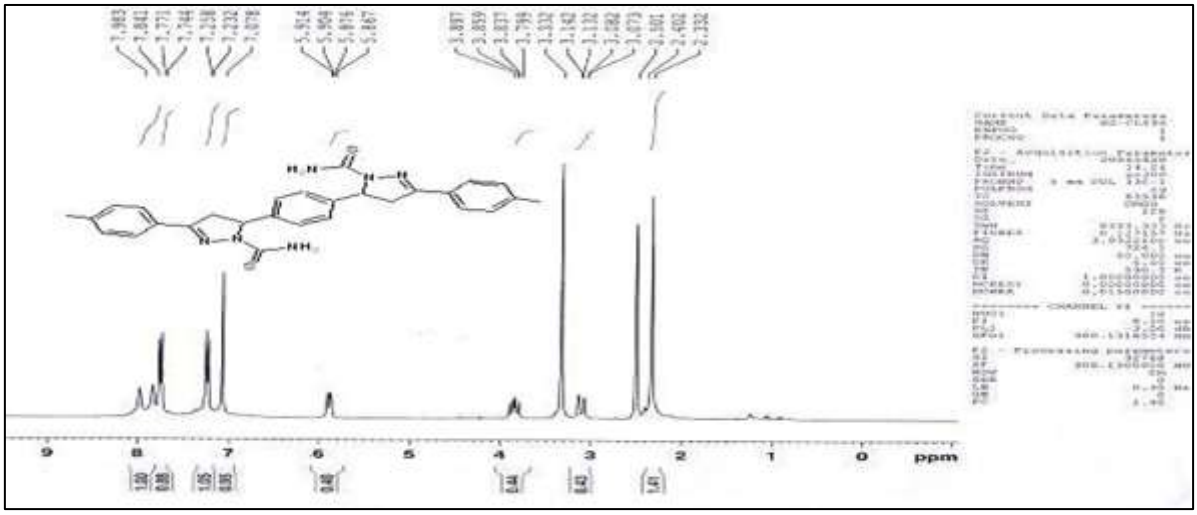


Figure (4): ^{1}H-NMR spectrum of compound (5l)

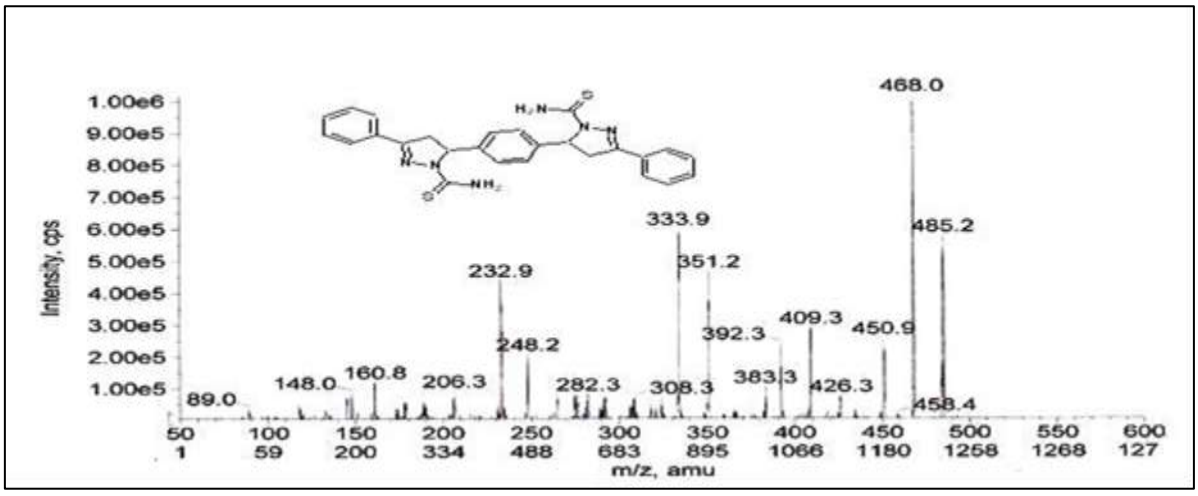


Figure (5): Mass spectrum of compound (5k)

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ئامادەکردن و دیاریکردنی هەندێ ئاوێتەی دووانە چالکون و گۆڕانیان بۆ ئاوێتەی نوێی تایوکاربامیل-2- بایرازولین

پوختە

ماددە سەرەتاییەکانی(1a-j, 2 and 3) ئەم توێژینەوەیە، ئامادەکران بە ڕێگای وولیامسن. ئەمانە و هەندێك ماددەی تری سەرەتایی کارلێکیان پێکرا لەگەل تیرفتال ئەلدیهاید و بە ڕێگای جیاوازی وەك: شەپۆلی سەرووی دەنگ و مایکرۆیف و ڕێگای بێ توێنەر، بۆ بە دەست هێنانی ئاوێتەی نوێی دوانە چالکۆنی (4a-s) . چالکۆنە ئامادە کراوەکان کارلێکیان پێکرا لەگەل تایوسیمی کاربازاید بە مە بەستی دروست کردنی ئاوێتەی نوێی تایوزیلیدینونی (5a-s).شێووگی کیمیاوی ماددە بەرهەم هاتوەکان دیاری کران بە بەکارهێنانی ڕێگای شەبەنگەکان وەك شەبەنگی ژێر سوورو(IR)و شەبەنگی لەرینەوەی ناوکی موگناتیسی بە هەر سی جۆری (^{1}H-NMR, ^{13}C-NMR, ^{13}C-DEPT-135)و شەبەنگی بارستە.

تحضير و دراسة بعض مركبات الجالكون و تحويلها الى مشتقات ثايوكاربامايل-2- بايرازولين الجديدة

الخلاصة

المركبات البادئة(1a-j, 2 and 3) في هذا البحث، تم تحضيرها بطريقة ووليلمسن. هذه المركبات و مع سلسلة اخرى من مواد بادئة مختبرية تفاعلت مع تيرفثالالديهابد و طرائق مختلفة مثل: الموجات الفوق الصوتية و المايكروبف و بدون استخدام المذيب بهدف تحضير مركبات ثنائى-جالكون الجديدة (4a-s). تم تفاعل الجالكونات الجديدة مع ثايوسيمي كاربازايد لتحضير مركبات ثايوزيليدينون (5a-s) الجديدة .تم تشخيص تراكيب المركبات المحضرة بواسطة الطرق الطيفية .مثل طيف الاشعة تحت الحمراء (IR) والرنين النووي المغناطيسي بانواعها الثلاثة (^{1}H-NMR, ^{13}C-NMR, ^{13}C-DEPT-135)وطيف الكتلةMS).