Synthesis of Some New Heterocyclic Compounds Via Chalcone Derivatives

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Abstract

The chalcones 1(a,b) were prepared by the reaction of 2- acetyl benzofuran with two aromatic aldehydes in the presence of alkaline media. These chalcones are used as starting material to obtain the desired heterocyclic: pyrazolin, isoxazoline, pyrimidinthion, pyrimidinone, cyclohexanone and indazole derivatives. The structure of newly synthesized heterocyclic compounds were established on the basis of their melting points, elemental analysis(C.H.N), FTIR and ^{1}HMNR (for some of them) spectral data . The synthesized compounds have been screened for their antibacterial activities, they exhibited good antibacterial activity against Escherichia coli (G-) and Staphylococus aureus (G+) .

Key Words: chalcones, pyrazoline, isoxazoline, pyrimidinone, pyrimidinthion cyclohexanone and indazole.

Introduction

Chalcones represent an essential group of natural as well as synthetic products and some of them have wide range of pharmmacological activity such as anti-inflammatory, anti-fungal, antibacterial and anti-oxidnt agents [1-3]. Chalcones were used to afford pharmacologically-interesting heterocyclic systems like pyrazolines, isoxazoles and pyrimidines, cyclohexanone .

Pyrazolines for example are known to have anti-bacterial[4], anti-tumor[5], anti-histaminic[6] and anti-convulsant activity[7] , while isoxazole exhibit anti-microbial, anti-cancer[8] and analgesic activity[9].

Pyrimidines are associated with various biological activities like anti- inflammatory and analgesic activity[10]. Indole derivatives are reported to possess antibacterial, anti -cancer, anti-inflammatory[11] . Hence, it appeared of interest to prepare new derivatives of the mentioned nucleouses.

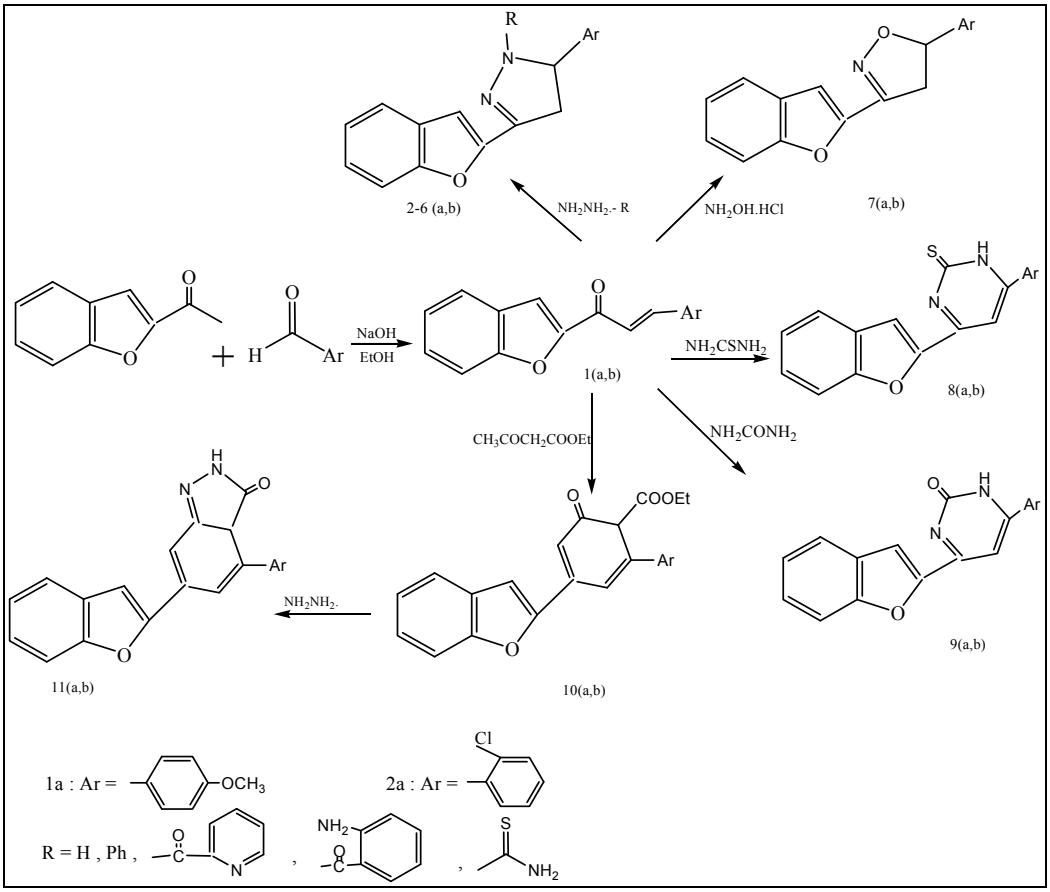
Experimental

Materials

All chemicals were supplied from Merck , Fluka and Aldrich Chemicals Co. and used as received .

Techniques

FTIR spectra were recorded using potassium bromide discs on a Shimadzo (IR prestige-21) FTIR spectrophotometer . ^{1}HNMR spectra were carried out by company : Bruker , model: ultra shield 300 MHz , origin : Switzerland and are reported in ppm(δ), DMSO was used as a solvent with TMS as an internal standard . Elemental analysis (C.H.N.S-O) were carried out using an EuroEA Elemental Analyzser at (The Central Service Laboratory-College of Education For Pure Sciences Ibn Al- Haitham). Uncorrected melting points were determined by using Hot-Stage, Gallen Kamp melting point apparatus .


2-6 (a,b)
NH2NH2.- R
NH2OH.HCl
7(a,b)
NaOH
EtOH
1(a,b)
NH2CSNH2
8(a,b)
CH3COCH2COOEt
NH2CONH2
COOEt
9(a,b)
NH2NH2.
11(a,b)
10(a,b)
1a : Ar =
OCH3
2a : Ar =
Cl
R = H , Ph ,
NH2
NH2

Scheme(1)

Synthesis

Synthesis of chalcones 1-(Benzofuran-2-yl)-3-(substituted phenyl) prop-2-ene-1-one (1a,b) [1]

A mixture of aromatic benzaldehyde (0.01 mol) and 2-acetyl bezofuran (0.01 mol, 1.60 g) was dissolved in (10 mL) of ethanol, an aqueous soudium hydroxide solution (5 mL, 25%) was added. The mixture was stirred for 2-3hrs. at room temperature. The mixture was kept in a refrigerator for overnight when it became quite thick. Then it was diluted with ice-cold distilled water (40 mL), filtered, washed well with cold water, dried in air and recrystallized from ethanol to give the required product (2.26 g, 82%), mp 118-120ºC.

Synthesis of 3-(Benzofuran-2-yl) Pyrazoline derivatives 2-6(a,b)

A mixture of chalcone 1(a,b) (0.01 mol) and hydrazine hydrate 99% (5mL, 0.01 mol) or substituted hydrazine hydrate (0.01 mol) in absolute ethanol (10 mL) containing glacial acetic acid (0.5 mL) was refluxed for 2h[2]. After cooling, the solid formed was filtered off, air dried and recrystallized form absolute ethanol. The physical data of these compoundes are listed in Table 1. Anal. Calcd for (4a) $C_{24}H_{18}N_3O_3$: C, 72.72 ; H, 4.54; N, 10.60. Found: C, 73.23; H, 4.95; N, 9.87.

Synthesis of 3-(Benzofuran-2-yl)isoxazoline derivatives 7(a,b) [8]

To a mixture of chalcone (0.01 mol) and hydroxyl amine hydrochloride (0.01 mol , 0.69 g), absolute ethanol (50mL), aqueous sodium hydroxide (10%, 6 mL) were added then the reaction mixture was heated under reflux for 8h and poured slowly into ice cold water and the product obtained was washed with water and recrystallized from absolute ethanol. The physical data of these compoundes are listed in Table 1. Anal. Calcd for (7b) $C_{17}H_{12}NO_2Cl$: C, 68.45; H, 4.02; N, 4.69. Found: C, 67.95; H, 3.87; N, 4.02.

Synthesis of Pyrimidinethion derivatives 8(a,b) [5]

To a solution of chalcone (0.01 mol) absolute ethanol (10 mL), thiourea (0.01 mol, 0.6 g) and aqueous sodium hydroxide (10 mL, 20.0 mmol) were added. The reaction mixture was heated under reflux for 7h and poured into iced cold water the product obtained was filtered, washed with water and recrystallized from absolute ethanol . The physical data of these compoundes are listed in Table 1. Anal. Calcd for (8b) $C_{18}H_{11}N_2OSCl$: C, 63.71; H, 3.24; N, 8.25; S, 9.43. Found: C, 62.95; H, 3.65; N, 7.93 ;S,9.10.

Synthesis of Pyrimidinone derivatives 9 (a,b) [5]:

To a solution of (0.01 mol) of chalcone, absolute ethanol (10 mL), urea (0.01 mol, 0.6 g) of aqueous sodium hydroxide (10mL, 10 %) were added. The reaction mixture was heated under reflux for 5h and poured in ice-cold water. The product obtained was filtered washed with water and recrystallized from ethanol (95%) . The physical data of these compoundes are listed in Table 1.

Synthesis of Cyclohexanone derivatives 10(a,b) [11] :

A mixture of chalcones (1a,b) (0.01 mol) and ethyl acetoacetate (1.30mL, 0.01mol) in absolute ethanol (10mL) containing aqueous potassium hydroxide solution (1 mL, 10%) was refluxed for 5h and then left overnight at room temperature. The solid formed was filtered off, air dried and recrystallized form absolute ethanol. The physical data of these compoundes are listed in Table 1. Anal. Calcd for (10a) $C_{24}H_{20}O_5$: C, 74.22; H, 5.15.. Found: C, 74.87; H, 5.85.

Synthesis of Indazole derivatives 11(a,b) [11]

A mixture of compounds10(a,b) (0.01 mol) and hydrazine hydrate 99% (5mL, 0.01 mol)in absolute ethanol (10 mL) containing glacial acetic acid (0.5 mL) was refluxed for 2h. After cooling, the solid formed was filtered off, air dried and recrystallized form chloroform. The physical data of these compoundes are listed in Table 1. Anal. Calcd for (11a) $C_{22}H_{17}N_2O_3$: C, 73.94; H, 4.76; N, 7.84. Found: C, 73.11; H, 3.99; N, 7.21.

Result and Discusion

The synthesis of chalcones, pyrazoline, isoxazoline, pyrimidin -thion, pyrimidinon, cyclohexanone and Indazole derivatives were performed as shown in scheme (1).

The starting chalcones, namely 1-(benzofuran-2-yl)-3-(4-methoxyphenyl) prop-2-ene-1-one (1a) and 1-(benzofuran-2-yl)-3-(2-chlorophenyl) prop-2-ene-1-one (1b), were synthesized via the Claisen- Schmidt reaction of 2-acetyl benzofuran with 4- methoxybenzaldehyde and 2-chlorobenzaldehyde, respectively, in ethanol and in the presence of aqueous soudium hydroxide at room temperature. The structural assignments of the chalcones 1(a,b) based on melting points and FTIR spectroscopy.

The FTIR spectrum of chalcone (1a) indicated the appearance of two peaks at 1655cm^{-1}and1573cm^{-1}due to of C=O and C=C stretching vibrations ,respectively. peak at 1163 due to C-O-C . Also the FTIR spectrum of chalcone (1b) indicated the appearance of two peaks at 1666cm^{-1}and1612cm^{-1}due to of C=O and C=C stretching vibrations ,respectively.

Reaction of chalcones 1 (a,b) with hydrazine hydrate, phenyl hydrazine, 2-pyridinecarboxylic acid hydrazide and 2-aminobenzo- hydrazide under reflux in the presence of glacial acetic acid to yield the corresponding pyrazoline derivatives 2-6(a,b), respectively.

The structure of the pyrazoline derivatives 2-6(a,b) was identified by their melting point, C.H.N analysis, FTIR and ^{1}HNMR spectroscopy. The FTIR spectra of these compounds showed the disappearce of two absorption bands of the CH=CH and C=O group , in the chalcone 1(a,b) and appearance of new absorption stretching bands of NH and C=N groups (Table 2). ^{1}HNMR spectrum of compound (2b), (Figure 1), (in DMSO as a solvent) shows the following signals: a sharp singlet signal at δ 2.49ppm due to a proton of N-H group , sharp signals at δ 3.37ppm could be attributed to two protons of CH_2 group- pyrazoline, a signal in the region δ 7.35ppm for furan ring conjugate with benzene ring, many signals(aromatic protons) appeared in the region δ 7.81-7.95 ppm.

Isoxazoline compounds 7(a,b) were synthesized from the reaction of chalcones 1(a,b) with hydroxylamine hydrochloride in alkaline medium. The FTIR spectra of isoxazoline 7(a,b) showed the disappearance of two absorption bands of the CH=CH and C=O group in the starting material together with the appearance of new absorption bands for C=N group around 1610 $cm^{-1}$and C-O (cyclic ether) group around 1178 cm^{-1}. The FTIR spectral data for isoxazoline 7(a,b) are listed in Table(2).

^{1}HNMR spectrum of compound (7a) , Figure (2), (in DMSO as a solvent), showed many signals(protons of aromatic protons) appeared in the region δ 7.04-8.25 ppm and a signal in the region δ 6.78ppm for furan ring conjugate with benzene ring. The triplet signal at δ 3.82ppm and a doublet signal at δ 3.68ppm due to one protons C-5 and two protons C-4 in the isoxazoline ring, respectively. Furthermore, a sharp signal at δ 3.56ppm for three protons of OCH_3 group.

Pyrimidinethion derivatives 8(a,b) were synthesized from the reaction of chalcones1(a,b) with thiourea in basic medium .The structure of the compounds 8(a,b) are characterized by FTIR and ^{1}HNMR spectroscopy.The characteristic FTIR adsorption band of

pyrimidinethion showed the disappearance of two absorption bands of the CH=CH and C=O groups in the chalcones and appearance of new absorption bands for NH and C=S groups around 3384cm^{-1}and 1136cm^{-1}, respectively. The FTIR spectral data of these compounds are shown in Table 2 . ^{1}HNMR spectrum of pyrimidinethion (8a) , Figure (3), exhibited eight aromatic protons appeared as many pairs of doublet at δ 6.99-7.91ppm, a singlet signal at δ6.785ppm for furan ring conjugate with benzene ring, a singlet doublet at δ 6.780ppm for proton at C-5 of pyrimidinethion ring. A sharp singlet at δ 3.36ppm due to three protons of OCH_3 group. a singlet at δ 2.51ppm that could be attributed to one proton of NH group .

The pyrimidinone derivatives 9 (a,b) were synthesized from reaction of chalcone 1(a,b) with urea in basic medium. The structure of the pyrimidinone 9(a,b) characteristic by FTIR spectra which showed the disappearance of two absorption bands and appearance of new absorption bands for NH and C=O groups around 3338cm^{-1}and 1665cm^{-1}, respectively. The other data of functional groups which are characteristic of these compounds are given in Table 2. ^{1}HNMR spectrum of compound (9b), Figure (4), shows the following signals: eight aromatic protons appeared at δ (7.07-7.88) ppm, a singlet signal at δ 6.75 ppm could be attributed to the one proton of C-5pyrimidinone and a singlet at δ 5.15 ppm due to the proton of C6-pyrimidinone. Allso, singlet broad signal one proton of NH group appeared as δ3.36 ppm .

The chalcones 1(a,b) were allowed to react with ethyl acetoacetate (1:1) in the presence of aqueous potassium hydroxide10% to give new cyclohexanone derivatives 10 (a,b). The structure of the cyclohexanone 10(a,b) was identified by their melting point, C.H.N analysis, FTIR and ^{1}HNMR spectroscopy . The FTIR spectral data of these compounds are shown in Table 2. ^{1}HMNR spectrum of compound (10b), shows the following signals: eight aromatic protons appeared in the region δ 7.01-7.87 ppm . The quartet singnal at δ 4.42ppm that are attributed to two protons of CH_2 CO groups and a triplet signal at δ 1.61 ppm is due to three protons of CH_3CH_2CO. Four protons of $CHCHCH_2$ cyclohexanone appears as multiplet at δ 3.55 ppm .

The reaction may have proceeded through condensation between C=O of cyclohexanone and NH_2 of hydrazine, followed by cyclization by losing a molecule of ethanol. The new indazole derivatives 11(a,b) were synthesized by refluxing compounds 10(a,b) and hydrazine in the presence of glacial acetic acid. The FTIR spectra of these compounds showed the disappearce of absorption bands and appearance of new absorption bands of NH and C=N group at 3363cm^{-1} and 1603cm^{-1}, respectively. Functional groups which are characteristic of these compounds are given in Table 2. Finally, the ^{1}HNMR spectrum of compound (11a) (in DMSO), shows the following signals: eight aromatic protons appeared in the region δ 7.20-8.73 ppm. a singlet signal at δ 6.80ppm for furan ring conjugate with benzene ring. a signal at δ 3.12 ppm that are attributed to cyclohexanone protons . Furthermore, a singlet signal at δ 2.93ppm which was assigned to proton of NH .

Biological Activity

All the synthesized compounds were tested for their antimicrobial activity against Gram negative bacteria (Escherichia Coli) and Gram positive bacteria (Staphylococcus aureus) using the agar diffusion method [11]. Each compound was dissolved in DMSO to give concentration 1ppm. The plates were then incubated at 37 ^{0}C and examined after 24 hrs. The zones of inhibition formed were measured in millimeter and are represented by (-),(+), (+ +) and (+ + +) depending upon the diameter and clarity as in Table 3. All the compounds exhibit the highest or lower biological activity against both of the organisms .

All compounds showed good inhibition against the two types of the bacteria, this could be related to the presence of heterocyclic rings .

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Table No. (1): Physical properties of synthesized compounds 2-11(a,b)

Comp. No.	Namecalture	Molecular Formula	M.P ^{0}C	Yield %	Color
2a	4,5-dihydro-3-(1-benzofuran-2-yl)-5-(4-methoxyphenyl) -1H- pyrazole	$C_{18}H_{15}N_2O_2$	180-182	78	Pale yellow
2b	4,5-dihydro-3-(1-benzofuran-2-yl)-5-(2-chlorophenyl) - 1H- pyrazole	$C_{17}H_{12}N_2OCl$	91-93	72	Yellow
3a	4,5-dihydro- 3-(1-benzofuran-2-yl)-5-(2-methoxyphenyl) -1-phenyl-1H- pyrazole	$C_{24}H_{19}N_2O_2$	223-225	68	Pale Brown
3b	4,5-dihydro-3-(1-benzofuran-2-yl)-5-(2-chlorophenyl) -1-phenyl-1H- pyrazole	$C_{23}H_{16}N_2OCl$	174-176	67	Orange
4a	4,5-dihydro-3-(1-benzofuran-2-yl)-5-(4-methoxyphenyl) -1 (2-pyridine carboxylic acid) -1H- pyrazole	$C_{24}H_{19}N_3O_3$	124-126	77	Yellowish Brown
4b	4,5-dihydro-3-(1-benzofuran-2-yl)-5-(2-chlorophenyl -1 (2- pyridine carboxylic acid)-1H-pyrazole	$C_{23}H_{14}N_3O_2Cl$	89-91	75	Red
5a	4,5-dihydro- 3-(1-benzofuran-2-yl)-5-(4-methoxyphenyl) -1(2-aminobenzo) -1H-pyrazole	$C_{25}H_{21}N_3O_3$	200-202	60	Pale yellow
5b	4,5-dihydro-3-(1-benzofuran-2-yl)-5-(2-chlorophenyl) -1(2-aminobenzo -1H-pyrazole	$C_{24}H_{18}N_3O_2Cl$	138-140	68	Brownish Yellow
6a	4,5-dihydro- 3-(1-benzofuran-2-yl)-5-(4-methoxy phenyl) pyrazole-1-carbothioamide	$C_{19}H_{16}N_3O_2S$	244-246	76	Off-white
6b	4,5-dihydro- 3-(1-benzofuran-2-yl)-5-(2-chlorophenyl) pyrazole-1-carbothioamide	$C_{18}H_{13}N_3OClS$	228-230	72	White
7a	3-(1-benzofuran-2-yl)-5-(4-methoxy phenyl)-4,5-dihydroisoxazole	$C_{18}H_{15}NO_3$	83-85	70	Yellowish brown
7b	3-(1-benzofuran-2-yl)-5-(2-chloro phenyl)-4,5-dihydroisoxazole	$C_{17}H_{12}NO_2Cl$	151-153	66	bright Brown
8a	6-(1-benzofuran-2-yl)-4-(4-methoxy phenyl) pyrimidine- 2(1*H*)- thione	$C_{19}H_{14}N_2O_2$ S	116-118	76	Brown
8b	6-(1-benzofuran-2-yl)-4-(2-chloro phenyl) pyrimidine- 2(1*H*)- thione	$C_{18}H_{11}N_2OClS$	90-92	75	Brownish yellow
9a	6-(1-benzofuran-2-yl)-4-(4-methoxy phenyl) pyrimidin -2(1*H*)- one	$C_{19}H_{14}N_2O_3$	174-176	60	Yellowish Brown
9b	6-(1-benzofuran-2-yl)-4-(2-chloro phenyl) pyrimidin -2(1*H*)- one	$C_{18}H_{11}N_2O_2Cl$	183-185	64	Light brown
10a	Ethyl-4-(1-benzofuran-2-yl)- 6- (4-methoxyphenyl)-2-oxocyclohexa-3-enecarboxylate	$C_{24}H_{20}O_5$	66-68	78	Green-Yellow
10b	Ethyl-4-(1-benzofuran-2-yl)- 6- (2-chlorophenyl)-2-oxocyclohexa-3-enecarboxylate	$C_{23}H_{17}O_4$ Cl	127-129	65	Pale Brown
11a	4,5-dihydro-4-(4-methoxyphenyl)- 6-(1-benzofuran-2-yl)-2H-indazol-3(H)ones	$C_{22}H_{17}N_2O_3$	75-77	60	Off-white
11b	4,5-dihydro-4-(2-chlorophenyl)- 6-(1-benzofuran-2-yl)-2H-indazol-3(H)ones	$C_{21}H_{14}N_2O_2$ Cl	113-115	50	Gray

Table No.(2):Characteristic FTIR absorption bands of synthesized compounds 2-11(a,b)

Comp. No.	ν N-H	ν C=O	ν C=N	ν C=C	ν C=C aromatic	ν C=S	Others
2a	3394	-	1653	1610	1558	-	C-OCH_3: 831
2b	3441	-	1622	1600	1554	-	C-Cl:750
3a	-	-	1620	1595	1554	-	C-OCH_3: 829
3b	-	-	1644	1597	1559	-	C-Cl:750
4a	-	1664	1654	1593	1573	-	C-OCH_3: 819
4b	-	1662	1632	1601	1558	-	C-Cl:752
5a	-	1680	1654	1592	1573	-	C-OCH_3: 820
5b	-	1666	1612	1591	1551	-	C-Cl:750
6a	-	-	1610	1585	1549	1249	C-OCH_3: 829
6b	-	-	1640	1574	1545	1257	C-Cl:751
7a	-	-	1653	-	1589	-	C-O-N: 1251
7b	-	-	1647	-	1558	-	C-O-N: 1342
8a	3419	-	-	1671		1160	C-OCH_3: 830
8b	3384	-	-	1652		1136	C-Cl:751
9a	3384	1665	-	1620	1559	-	C-OCH_3: 819
9b	3444	1660	-	1643	1555	-	C-Cl:751
10a	-	1731	-	1650	1600	-	C-OCH_3: 836
10b	-	1743	-	1645	1592	-	C-Cl:750
11a	3363	1709	1602	1535	1519	-	C-OCH_3: 819
11b	3344	1713	1600	1590	1510	-	C-Cl:773

Table No. (3) : antibacterial activity of the synthesized compounds 2-11(a,b)

Comp. No.	E. Coli (G-)	Staphlococcus aurus(G+)	Comp. No.	E. Coli (G-)	Staphlococcus aurus(G+)
2a	++	+	7a	+++	+++
2b	++	++	7b	+++	++
3a	+	++	8a	++	+++
3b	+	++	8b	++	++
4a	++	+++	9a	+	++
4b	+++	++	9b	++	++
5a	++	+	10a	++	+
5b	++	+	10b	+	+
6a	+++	++	11a	+++	+++
6b	++	++	11b	+++	+++

Key to symbols: Highly active = + + +(mor than)15 mm. Moderately active = + +(11-15) mm and slightly active = + (5-10) .

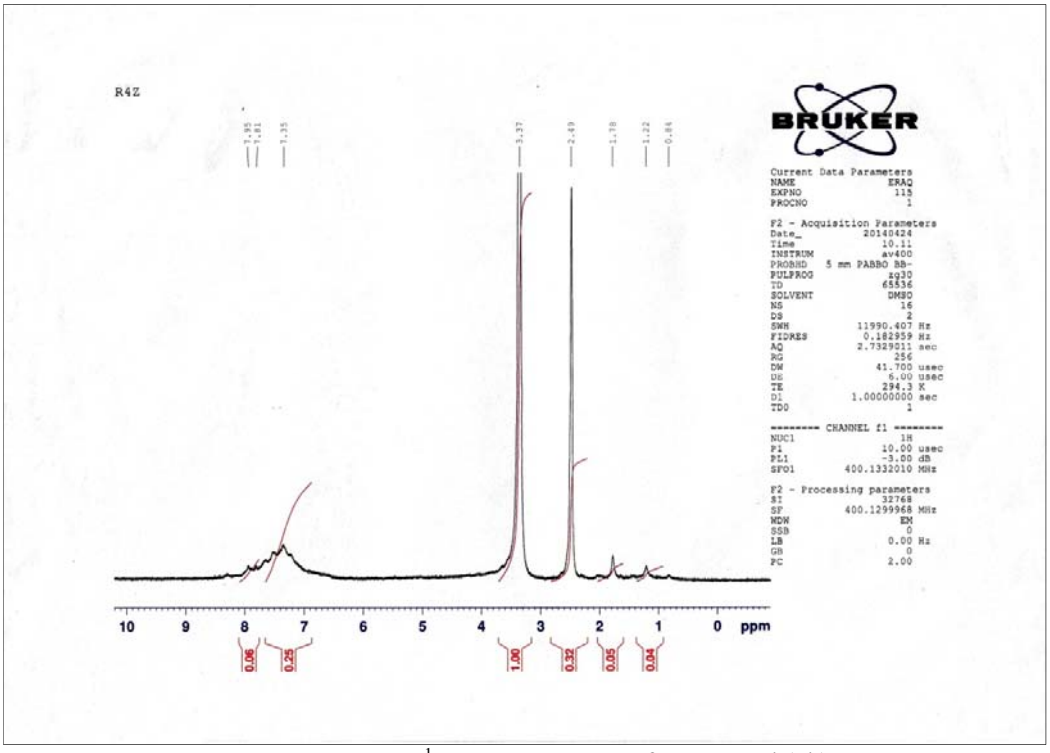

Figure No.(1):[1]HNMR spectrum of compound (2b)

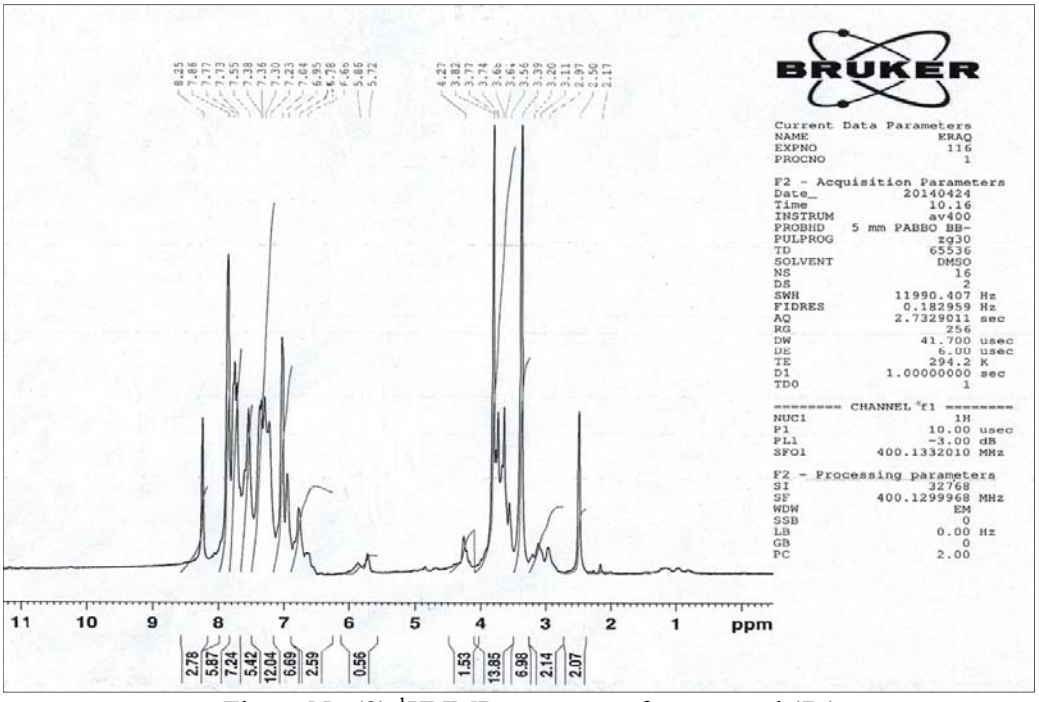

Figure No.(2):[1]HNMR spectrum of compound (7a)

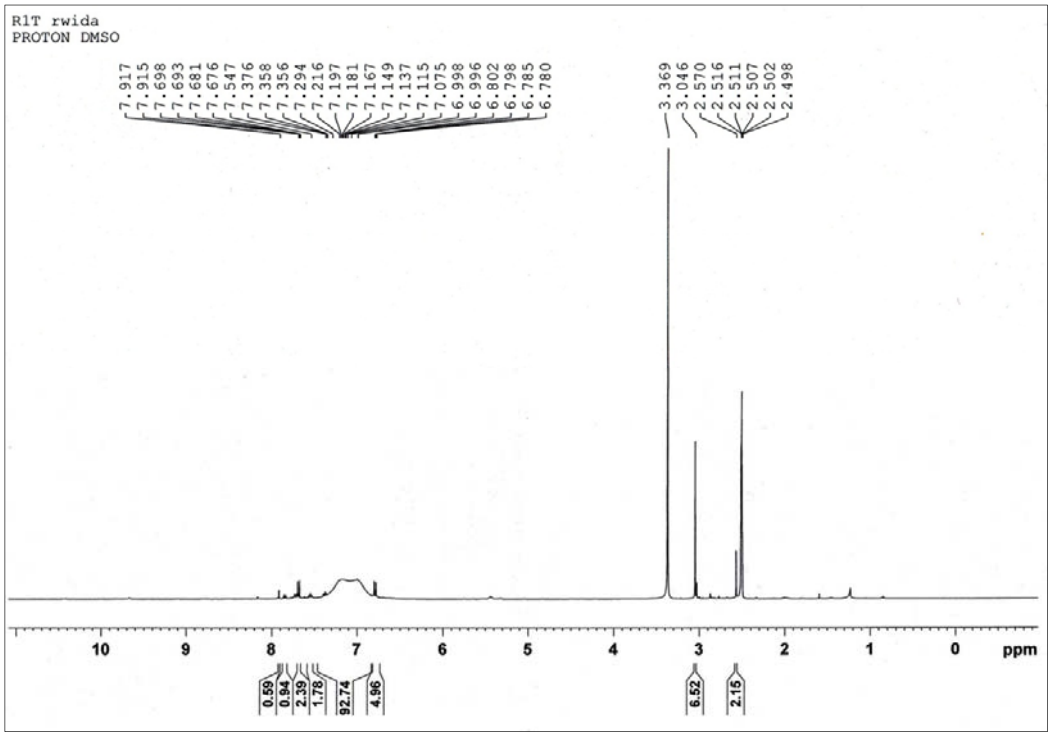

Figure No. (3):^{1}HNMR spectrum of compound (8a)

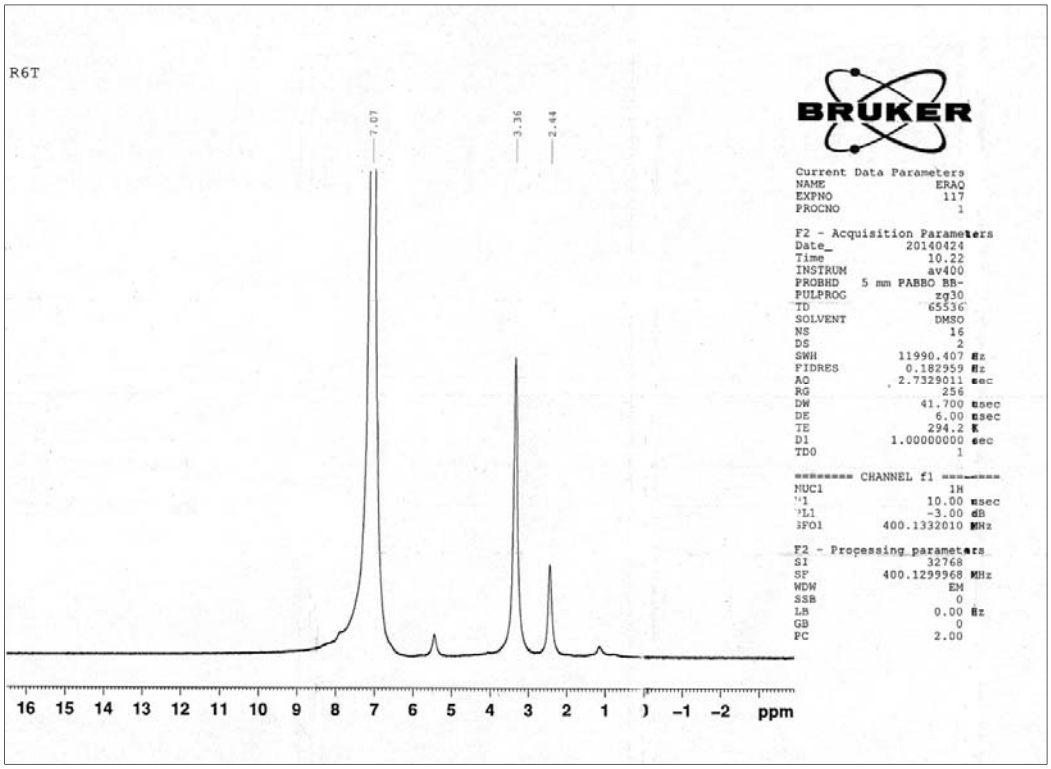

Figure No.(4):^{1}HNMR spectrum of compound (9b)

تحضير بعض المركبات الحلقية غير المتجانسة الجديدة من مشتقات الجالكون

منى سمير سعيد

قسم الكيمياء / كلية التربية للعلوم الصرفة(ابن الهيثم)/ جامعة بغداد

استلم البحث في:1 ايلول 2014،قبل البحث في :7 كانون الاول 2014

الخلاصة

حضرت الجالكونات 1(a,b) من تفاعل 2- استيل بنزوفيوران مع اثنين من الالديهايدات الاروماتية في وسط قاعدي . استعملت هذه الجالكونات مادة اولية للحصول على مشتقات حلقية غير متجانسة هي :البايروزولين , الايزوكزازولين , والبريميدينثايون , والبريميدينون , والسايكلوهكسانون , والاندازول . وشخص تركيب المركبات الحلقية غير المتجانسة الجديدة المحضرة عن طريق قياس درجات انصهارها وتحليل العناصر الدقيق للعناصر(C.H.N) والطرائق الطيفية FTIR و ^{1}HMNR (لبعضها). كما درست الفعالية البايولوجية للمركبات المحضرة واظهرت فعالية بايولوجية مختلفة ضد البكتريا بنوعيها Echerichia coli (G-) و Staphylococcus (G+) .

الكلمات المفتاحية:جالكون,بايرازولين,ايزكسوزولين,بريميدون,ثايوبريميدين,سايكلوهكسانون,اندازول.