Synthesis and Characterization New Schiff Bases,Pyrazole and Pyrazoline Compounds Derived From Acid Hydrazide Containing Isoxazoline Ring

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Abstract

The work involves synthesis of new Schiff bases ($[V]_{a, b}$ and $[VI]_{a, b}$), pyrazoles$[VII]_{a, b}$ and pyrazolines$[VIII]_{a, b}$ derivatives containing isoxazoline unit starting with chalcones. 4-bromoacetophenone was reacted with 4-hydroxybenzaldehyde or 4-hydroxyacetophenone was reacted with 4-bromobenzaldehyde in basic medium to give chalcone by Claisen-Schemidt reaction. The chalcons $[I]_{a, b}$ was reacted with hydroxylamine hydrochloride to form isoxazolines $[II]_{a, b}$. which were reacted with ethyl chloro acetate in basic medium to get ester compounds$[III]_{a, b}$.The condensation new ester$[III]_{a, b}$ with hydrazine hydrate80% yieldedacid hydrazide $[IV]_{a, b}$.The later compound refluxing with 4-substituted benzaldehyde in dry benzene to give Schiff bases($[V]_{a, b}$and $[VI]_{a, b}$)while the reaction of acid hydrazide $[IV]_{a, b}$ with acetylacetone or ethyl aceto acetate to get pyrazole$[VII]_{a, b}$, pyrazolone$[VIII]_{a, b}$,respectively.The synthesized compounds were characterized by melting points, FTIR, mass and ^{1}H NMR spectroscopy(of someof them).

Key Words:chalcones, Schiff bases, isoxazoline, pyrazole, pyrazoline**.**

Introduction

Chalcones were prepared by condensation of acetophenone with aromatic aldehydes in presence of basic medium [1]. The Chalcone derivatives are important intermediate and also act as precursor for the synthesis of novel cyanopyridines, pyrazolines, isoxazoles,pyrimidines and tetrazole [2]. Five-member heterocyclic compounds isoxazoline are important for pharmaceutical industry and material science due to their various applications. Isoxazoline are present in the structures of many natural products. In fact, isoxazoline have a broad spectrum of their biological and pharmacological activities [3-6].

Pyrazoles are one of the important members of heterocyclic compounds with two adjacent nitrogens in a five-membered ring system. Because of their aromaticity and wide application in pharmaceutical and material industry, they have gained significant interest among the scientist [7-10]. Also the pyrazoline showed a wide spectrum of biological activities such as anti-bacterial, antifungal, herbicidal and anti-choligenic [11-14]. In the view of the varied biological , pharmacological and industry applications, we have planned to synthesis some isoxazoline derivatives containing imine, pyrazole or pyrazoline unit.

Experimental

Chemicals

All chemicals were supplied by fluka, GCC, merck and sigma-Aldrich chemicals Co. and used as received.

Techniques

FTIR spectra were recorded using potassium bromide discs on a Shimadzu(Ir prestige -21) ^{1}HNMR spectra were carried out by company:Bruker,model: ultra-shield 400MHz,origin: Switzerland and are reported in ppm(δ) DMSO were used as a solvent with TMS as an internal standard,measurements were made at Chemistry Department, science and Technology University, Jordan. The mass spectrum was recorded on shimadzu model 6CMS QL 1000 EX, made in Japan. Uncorrected melting points were determined by using Hot-Stage,Gallen Kamp melting point apparatus.

Synthesis

New compounds are synthesized according to scheme1

Synthesis of (chalcones) 3-(4-hydroxyphenyl)(4`-bromophenyl)-2-propene-1-one[I]$_a$ and 3-(4`-bromophenyl)-1-(4-hydroxyphenyl)prop-2-en-1-one[I]$_b$

Equimolar quantities of 4-bromo or 4-hydroxy acetophenone (0.01 mol),and 4-bromo or4- hydroxy benzaldehyde (0.01 mol) were dissolved in minimum amount of alcohol. Sodium hydroxide solution (0.02 mol) was added slowly and the mixture became cold. Then the mixture was poured slowly into 400 mL of ice water with constant stirring and kept in refrigerator for 24 hrs. The precipitate obtained was filtered [15],washed and recrystallized from ethanol .

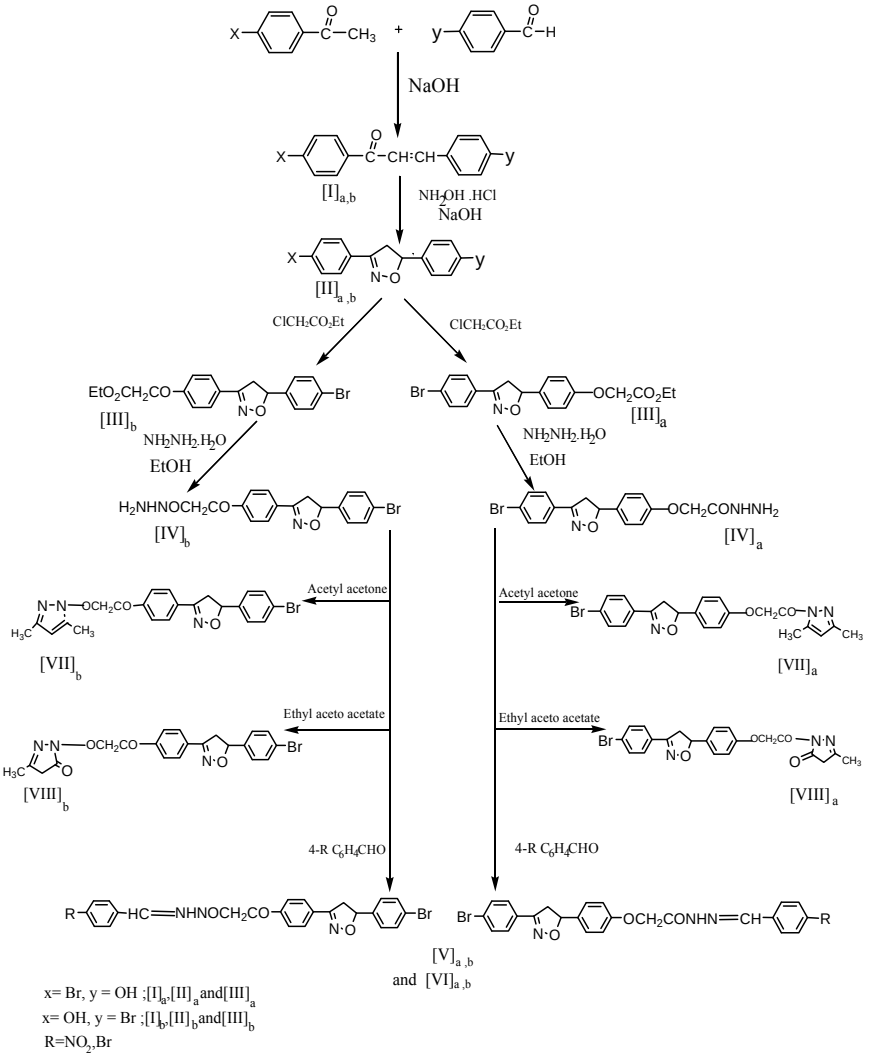
Synthesis of 4-(3-(4`-bromophenyl)-4,5-dihydroisoxazol-5-yl)phenol [II]$_a$ and 4-(5-(4`-bromophenyl)-4,5-dihydroisoxazol-3-yl)phenol[II]$_b$

A mixture of chalcone (0.02 mol) , hydroxylamine hydrochloride (0.02 mol , 1.39 gm) and sodium hydroxide solution (0.5 g NaOH in 25 mL of water) in ethanol (60 mL) was refluxed for 6hrs. The mixture was concentrated under vacuum and poured into ice water[16]. The precipitate obtained was filtered, washed and recrystallized from chloroform.

Synthesis of new ester derivatives[III]

A mixtuer of compound [II]$_{a, b}$(0.01mol) ,ethyl α- chloro acetate (0.01 mol) and fused sodium acetate (0.03mol,2.46gm) in ethanol 25mL was refluxed for 4 hrs .Then cooled and

poured into cold water, the resulting soiled was filtered and from recrystallized ethanol [17] to give a new ester.



Scheme (1)

Synthesis of hydrazid derivatives [IV]a, b

A solution of ester [III]a, b (0.06 mol) and hydrazine hydrate(15mL) in(25mL) of ethanol was heated to reflux during 4hrs. The mixture was then cooled to room temperature[18] ,and the solid obtained was filtered and recrystallized from ethanol.

The physical properties of synthesized compounds [I]-[IV] were given in Table 1.

Synthesis of Schiff base derivatives[V]a, b , [VI] a, b

A mixture of new hydrazide**[IV]a, b** (0.01 mol) , different aromatic aldehyde (0.012 mol) , dry benzene (10 mL) and 2drops of glacial acetic acid was refluxed for 3hrs . The solvent was evaporated under vacuum and the residue crystallized from chloroform[19].

Synthesis of pyrazole and pyrazoline derivatives [VII]a, b,[VIII]a, b

A mixture of new hydriozide **[IV]$_{a, b}$** (0.0028 mol) and $CH_3COCH_2COCH_3$or $CH_3COCH_2CO_2Et$ (0.0028 mol) in abs. EtOH(20mL) was refluxed for 3hrs. the reaction mixture was cooled and the formed precipitate was filtered off and recrystallized to give new pyrazoles [VII]$_{a, b}$ or pyrazoline[VIII]$_{a, b}$,respectively.
The physical properties of synthesized compounds [V]-[VIII] were listed in Table 2.

Results and Discussion

The chalcones [I]$_{a, b}$ were synthesized by Claisen-Schemidt reaction from condensation aromatic aldehyde with acetophenone in NaOH. The compounds [I]$_{a, b}$ were characterized by melting points, FTIR spectroscopy .The FTIR spectra of compound[I]$_{a, b}$ showed appearance broad band ν O-H between (3448-3250)cm^{-1}, absorption sharp stretching band in the region(1681-1645)cm^{-1} due to C=O stretching with the appearance band between (1654-1610) cm^{-1} due to ν C=C of chalcone unit and a stretching band at (680-675)cm^{-1}due to C-Br.Also the spectra showed disappearance characteristic bands of starting materials.

The isoxazoline compound [II]$_{a, b}$ was synthesized by the reaction of compound[I]$_{a, b}$ with hydroxylamine hydrochloride in basic medium .The FTIR spectra of compound [II]$_a$ showed disappearance the bands of C=O and C=C for chalcone moiety with the appearance of new bands for ν C-$H_{aliph.}$ in the region (2920-2854) cm^{-1} and appearance of a stretching band at (1643-1640) cm^{-1} due to ν C=N of isoexaline ring (endo cyclic) and ν C-O of isoexaline ring between (1095-1070) cm^{-1} .

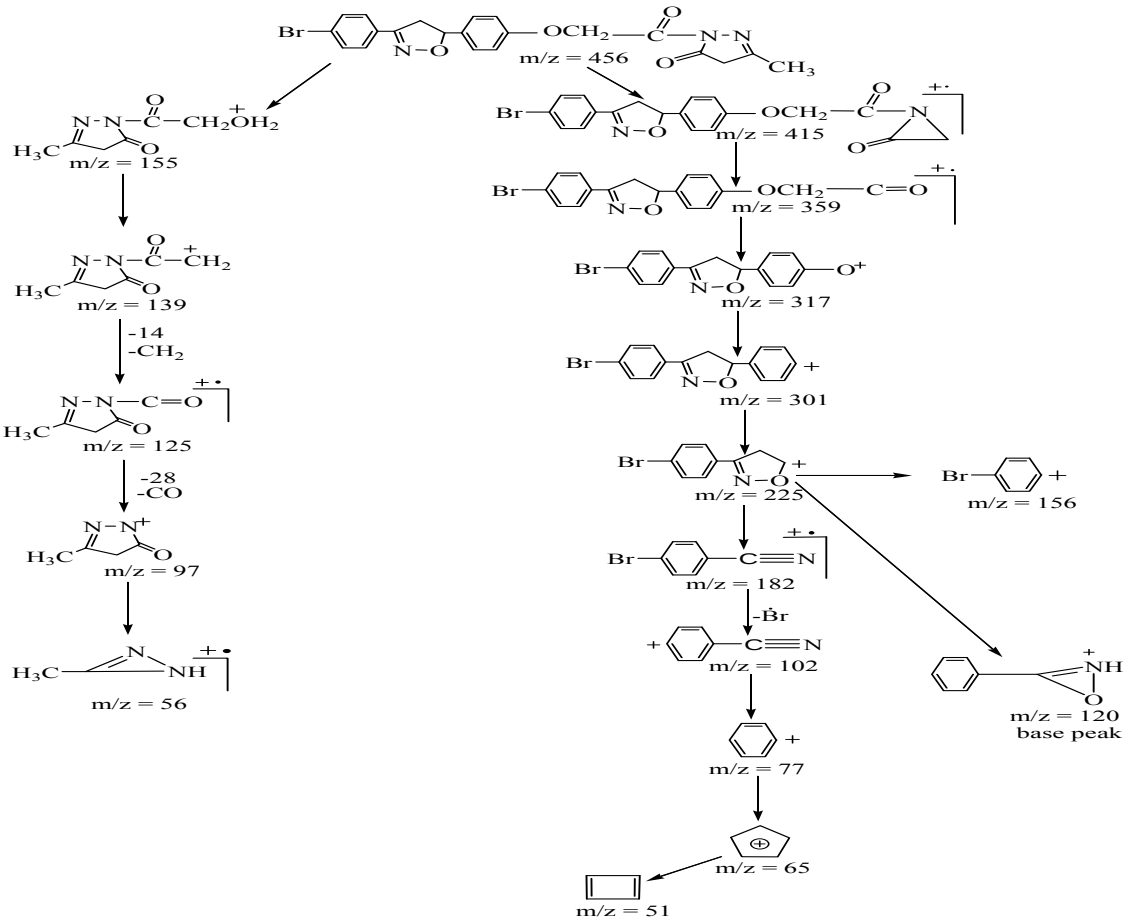
The ester compounds [III]$_{a, b}$ were synthesized by the reaction of compounds [II]$_{a, b}$ with ethylα-chloro acetate in fused sodium acetate. The FTIR spectra of compounds [III]$_{a, b}$ showed a significant band at 1735cm^{-1} which could be attributed to stretching vibration of the carbonyl of ester group ,together with disappearance absorption band due to ν O-H group for compound[III]$_{a, b}$ .The^{1}HNMR spectrum of ester compound [III]$_a$ (in DMSO as a solvent), Figure(1) showed the following characteristics chemical shifts: a singlet signal at δ4.25 ppm for two protons of OCH_2 group while the aquaterale signal of protons of CH_2 group appear at δ (3.12-3.20) ppm, and a triplet signal at δ1.79 ppm due to three protons of CH_3group. Also the spectrum showed many signals in the region δ (6.79-8.04) ppm could be attributed to eight aromatic protons , a triplet signal at δ (3.95-4.05) ppm and doublet of doublet at δ (2.95-3.02) ppm were assigned to one proton at C-5 and two protons at C-4,respectively of isoxazoline ring.

The condensation of ester with hydrazine hydrate to get new acid hydrazides[IV]$_{a, b}$. The FTIR spectra of compounds[IV]$_{a, b}$ showed a shift in the carbonyl stretching band to ester group of compound[III]$_{a, b}$ to 1645cm^{-1} for amide group of hydrazide[IV]$_{a, b}$ also showed three bands in the range(3334-3115) cm^{-1} which is assigned to asymmetric and symmetric bands of $NH_2$and NH groups. The^{1}HNMR spectrum of acid hydrazide[IV]$_b$ (in DMSO as a solvent), Figure(2) exhibited a sharp singlet at δ4.21 ppm for two proton of OCH_2 group, two signals at δ3.83 ppm and δ 4.46 ppm due to two protons at C-4 and one proton at C-5 ,respectively of isoxazoline ring. A broad signal at δ3.4 ppm could be assigned for two protons of NH_2 group and another broad signal at δ11.3 ppm due to proton of NH group. Finaly many signals between δ (6.63-7.87) ppm for eight aromatic protons.

The new Schiff bases compound [V]$_{a, b}$ and[VI] $_{a, b}$ were synthesized by the refluxing of compound [IV]$_{a, b}$ with different aromatic aldehydes in benzene. The compounds were characterized by melting points, FTIR spectroscopy . The characteristic FTIR absorption bands of compounds [V]$_{a, b}$ and[VI] $_{a, b}$ as Figure(3) showed the disappearance of two absorption bands due to NH_2 stretching of acid hydrazide together with the appearance of a

stretching bands at (1682-1671)cm^{-1} assignable to ν C=N The characteristics FTIR absorption bands of new Schiff bases $[V]_{a, b}$ and $[VI]_{a, b}$ were listed in Table 3.The refluxing hydrazide [IV] with acetyl acetone led to form new pyrazoles $[VII]_{a, b}$ .These compounds were characterized by melting points and FTIR spectroscopy. The FTIR spectra as Figure(4) showed the disappearance of three absorption bands due to NH_2 and NH groups together with the appearance of the stretching bands near (1645) cm^{-1} assignable to ν C=N group and 1371 cm^{-1} due to N-N for pyrazole ring .Also pyrazoline $[VIII]_{a, b}$ is produced from the reaction hydrazide with Ethyl aceto acetate. This compound is identified by melting points , FTIR spectra and mass spectroscopy .The FTIR spectra as Figure(5) showed the disappearance of three absorption bands due to NH_2 and NH groups together with the appearance of the a stretching band around 1650 cm^{-1} due to ν C=N band and new absorption band at (1735-1729)cm^{-1} due to C=O (endo cyclic) .

The mass spectrum of compound $[VIII]_a$ Figure(6) showed the most fragments in scheme 2.



Scheme (2)

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Table No. (1) The physical properties of Chalcones $[I]_{a, b}$ and compounds $[II]_{a, b}$-$[IV]_{a, b}$

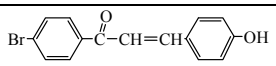
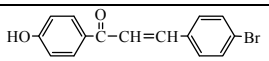
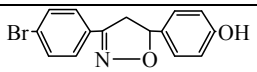
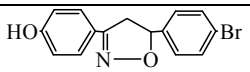
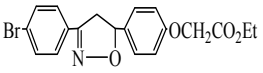
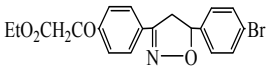
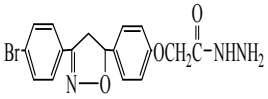
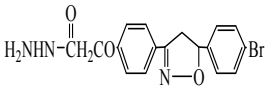
Com .No.	Nomenclature	Structural formula	Molecular formula	M. P ^0C	Yield %	Color
$[I]_a$	3-(4`-hydroxyphenyl)(4``-bromophenyl)-2-propene-1-one		$C_{15}H_{11}O_2Br$	158-160	45	Yellow
$[I]_b$	3-(4`-bromophenyl)-1-(4``- hydroxyphenyl) -2-propen-1-one		$C_{15}H_{11}O_2Br$	178-180	90	white
$[II]_a$	3-[4`-bromophenyl)-5-(4``-hydroxy phenyl)-4,5-dihydroisoxazole		$C_{15}H_{12}NO_2Br$	68-70	42	Yellow
$[II]_b$	3-[4`-hydroxy phenyl)-5-(4``-bromophenyl)-4,5-dihydroisoxazole		$C_{15}H_{12}NO_2Br$	130-132	60	Off white
$[III]_a$	Ethyl 2-{4-[3-(4`bromo phenyl)-4,5-dihydro isoxazol-5-yl]phenoxy} acetate		$C_{19}H_{18}NO_4Br$	100-102	59	Yellow
$[III]_b$	Ethyl 2-{4-[5-(4`-bromo phenyl)-4,5- dihydro isoxazol-3-yl]phenoxy} acetate		$C_{19}H_{18}NO_4Br$	155-158	70	Off white
$[IV]_a$	2-{4-[3-(4`-bromophenyl)-4,5-dihydroisoxazol-5-yl] phenoxy}acetohydrazide		$C_{17}H_{16}N_3O_3Br$	85-88	61	Pale green
$[IV]_b$	2-{4-[5-(4`-bromophenyl)-4,5-dihydroisoxazol-3-yl] phenoxy}acetohydrazide		$C_{17}H_{16}N_3O_3Br$	120-122	65	Pale green

Table No.(2) The physical properties of compounds $[V]_{a, b}$-$[VIII]_{a, b}$

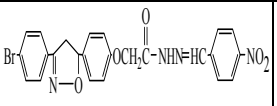
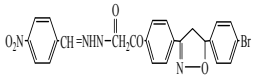
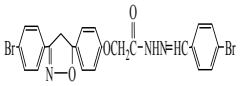
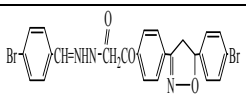
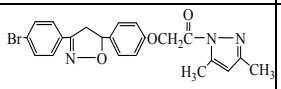
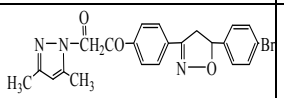
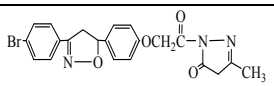
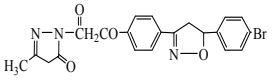
Com. No.	Nomenclature	Structural formula	Molecular formula	M. P ^0C	Yield %	Color
$[V]_a$	{4-[3-(4`-bromophenyl) -4,5-dihydro-isoxazol-5-yl]phenoxy}(4``-nitro benzylidine)acetic hydrazide.		$C_{24}H_{19}N_4O_5Br$	288-290	82	Brown
$[V]_b$	{4-[5-(4`-bromophenyl) -4,5-dihydro-isoxazol-3-yl]phenoxy}(4``-nitro benzylidine)acetic hydrazide.		$C_{24}H_{19}N_4O_5Br$	276-279	46	Yellow
$[VI]_a$	{4-[3-(4`-bromophenyl) -4,5-dihydro-isoxazol-5-yl]phenoxy}(4``-bromobenzylidine) acetichydrazide.		$C_{24}H_{19}N_3O_3Br_2$	158-160	82	Brown
$[VI]_b$	{4-[5-(4`-bromophenyl) -4,5-dihydro-isoxazol-3-yl]phenoxy}(4``-bromobenzylidine) acetichydrazide.		$C_{24}H_{19}N_3O_3Br_2$	110-114	92	Pale Brown
$[VII]_a$	2-{4-[3-(4`-bromo phenyl)-4,5-dihydroisoxazol-5-yl]phenoxy}-1-(3,5dimethyl-pyrazol -1-yl)ethanone.		$C_{22}H_{20}N_3O_3Br$	190-192	43	Brown
$[VII]_b$	2-{4-[5-(4`-bromo phenyl)-4,5-dihydro isoxazol-3-yl]phenoxy} -1-(3,5dimethyl-pyrazol -1-yl)ethanone.		$C_{22}H_{20}N_3O_3Br$	100-103	89	Brown
$[VIII]_a$	2(2-{4-[3-(4`-bromo phenyl)-4,5-dihydro-isoxazol-5-yl]phenoxy} -acetyl)-5-methyl -pyrazolin-3-one		$C_{21}H_{18}N_3O_4Br$	60-62	67	Brown
$[VIII]_b$	2(2-{4-[5-(4`-bromo phenyl)-4,5-dihydro-isoxazol-3-yl]phenoxy} -acetyl)-5-methyl -pyrazolin-3-one		$C_{21}H_{18}N_3O_4Br$	107-109	83	Brown

Table No.(3):Characteristic FTIR absorption band of compounds[V]$_{a, b}$ -[VIII]$_{a, b}$

Comp. No.	Characteristic bands FTIR spectra(cm^{-1})						
	νNH	νC-H aromatic	νC-H aliphatic	ν C=O endocyclic	ν C=O exocyclic	νC=C aromatic	Other
[V]$_a$	3271	3066	2957-2854		1662	1594	ν C=N 1671 νNO$_2$;1517,1390
[V]$_b$	3245	3077	2968-2843		1660	1595	ν C=N 1682 νNO$_2$;1521,1375
[VI]$_a$	3259	3065	2954-2843		1654	1589	ν C=N 1682
[VI]$_b$	3248	3071	2956-2854		1652	1588	ν C=N 1680
[VII]$_a$		3066	2954-2850		1675	1591	ν C=N(pyrazole) 1645
[VII]$_b$		3077	2954-2854		1665	1573	ν C=N(pyrazole) 1650
[VIII]$_a$		3066	2978-2854	1735	1712	1595	νC=N(pyrazoline) 1651
[VIII]$_b$		3077	2980-2861	1729	1711	1607	νC=N(pyrazoline) 1650

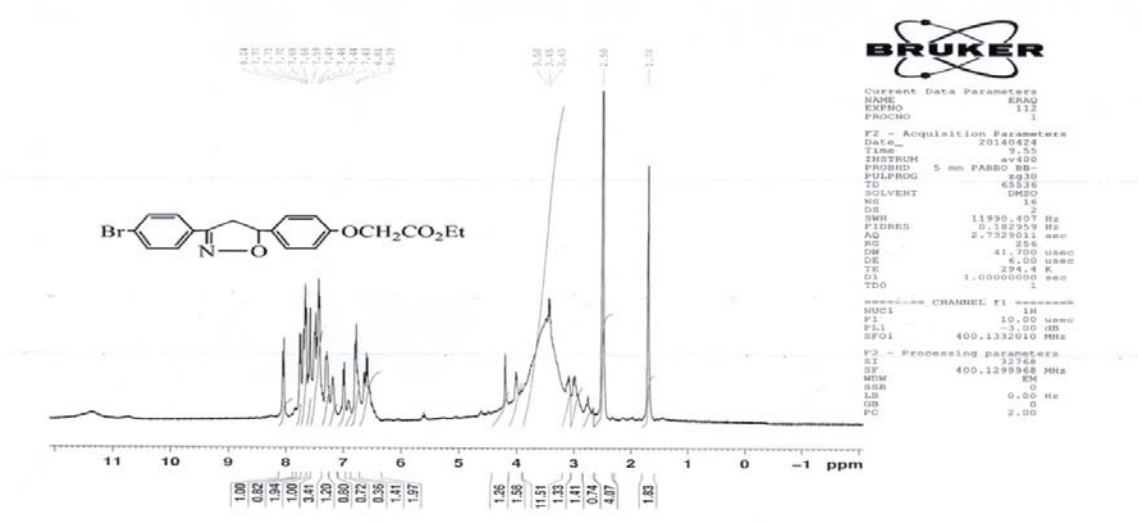


Figure No.(1): 1HNMR-Spectrum of compound

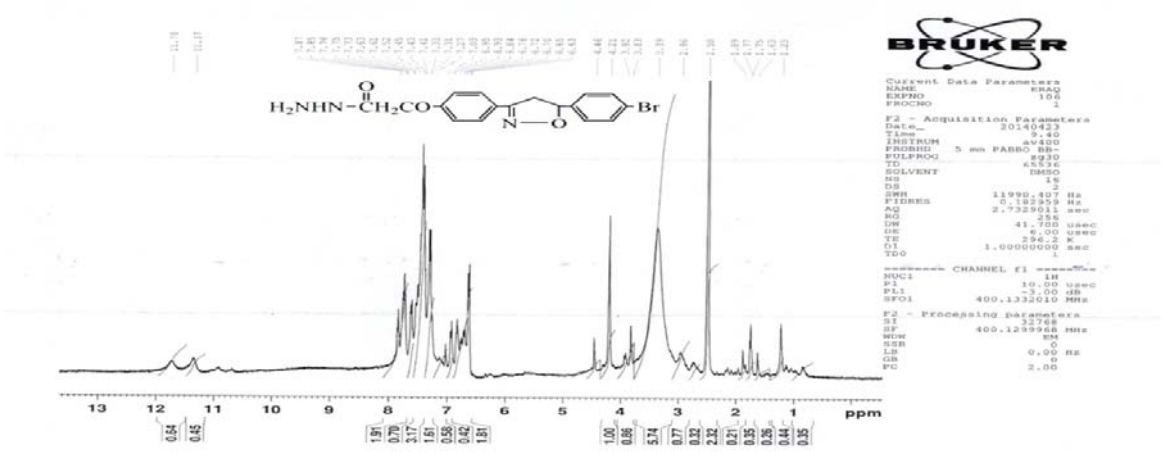


Figure No. (2) :1HNMR-Spectrum of compound $[IV]_b$

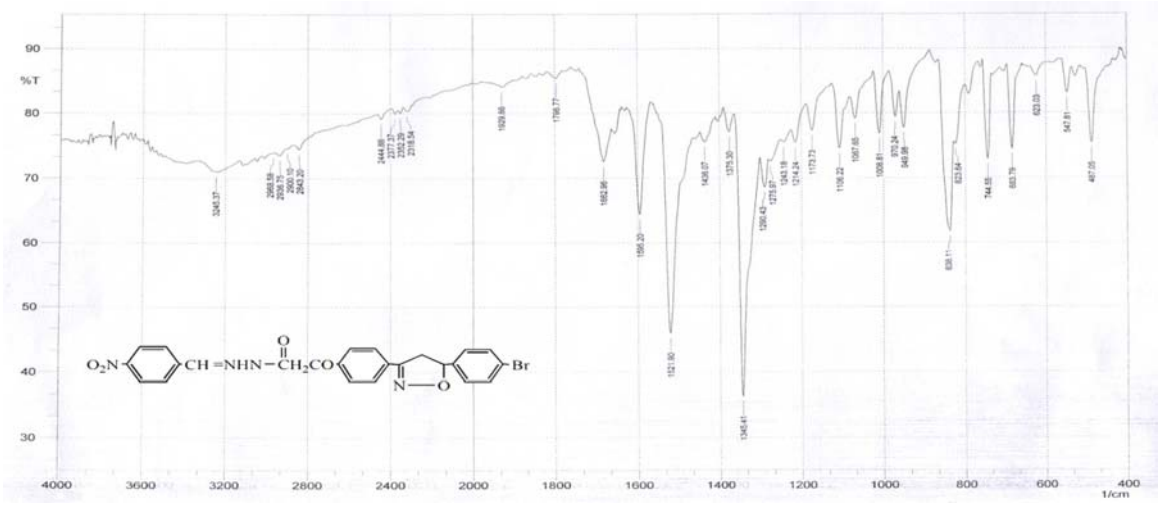


Figure No. (3): FTIR-Spectrum of compound $[V]_b$

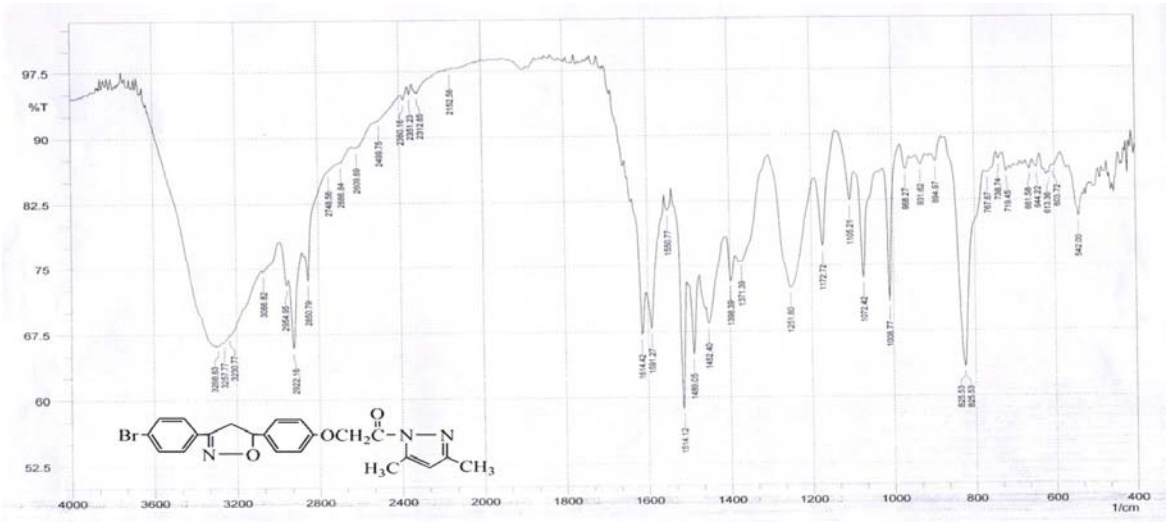


Figure No. (4) :FTIR-Spectrum of compound [VII]$_a$

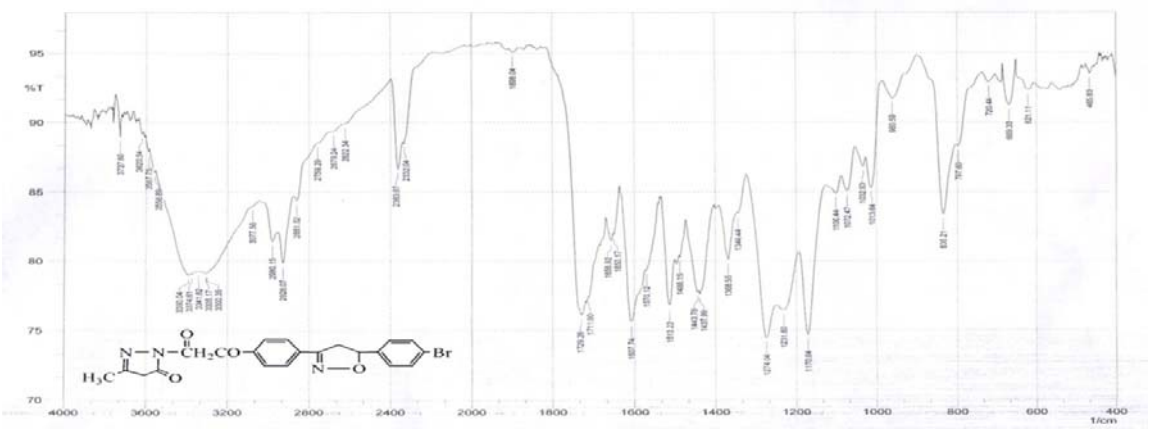


Figure No. (5): FTIR-Spectrum of cmpound [VIII]$_b$

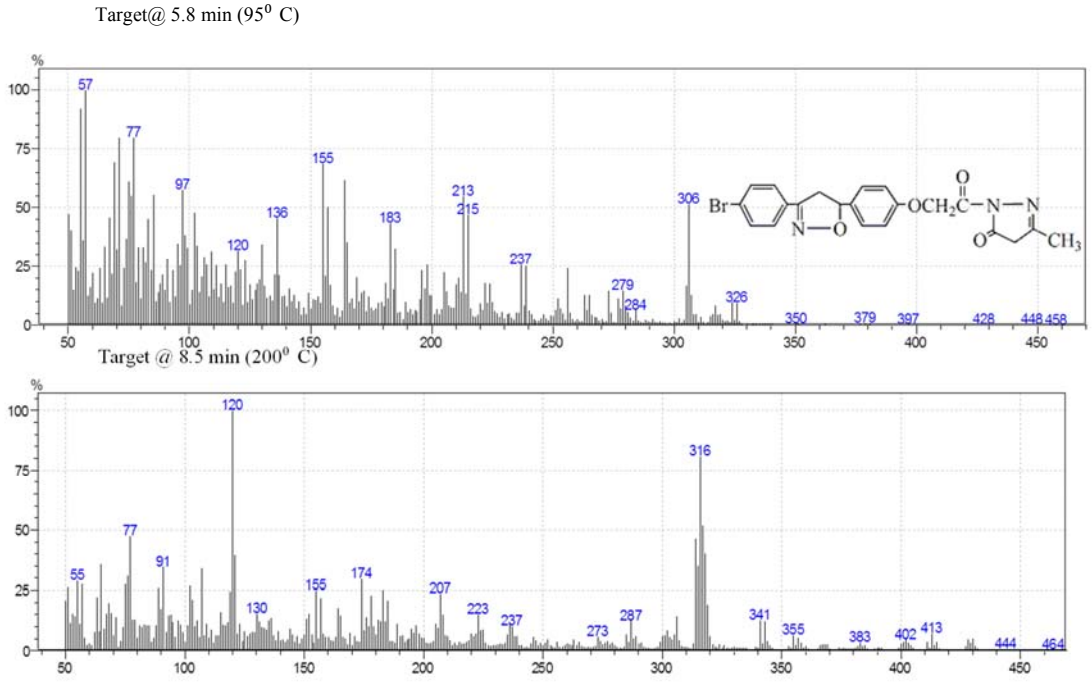


Figure No. (6): Mass - spectrum of compound [VIII]$_a$

تحضير و تشخيص مركبات قواعد شف و بايارازول و بايارازولين جديدة مشتقة من حامض الهيدرازيد الحاوي على حلقة الأيزوكزولين.

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الخلاصة

يتضمن هذا البحث تحضير مشتقات جديدة لقواعد شف$_{a, b}$ [V] و[VI]$_{a, b}$,بايرازول [VII]$_{a, b}$ وبايرازولين$_{a, b}$ [VIII] تحتوي على وحدةالايزوكزولين باستعمال الجالكون مادة أساسية ,يحضر الجالكون من تفاعل 4- برومو أسيتوفينون مع 4- هيدروكسي بنزالديهايد أوتفاعل 4- هيدروكسي أسيتوفينون مع 4- برومو بنزالديهايد في وسط قاعدي بتفاعل كلسين شمدت . يتفاعل الجالكون [I]$_{a, b}$ مع هيدروكسيل أمين هايدروكلورايد ليعطي الايزوكزولين [II]$_{a, b}$,الذي تمت مفاعلتة مع أثيل كلورو أسيتيت في وسط قاعدي لنحصل على مركب أستري [III]$_{a, b}$وحدة تكثيف الأستر الناتج مع الهايدرازين نحصل على حامض الهايدرازايد$_{a, b}$[IV]الذي يصعد عكسيا مع البنزلديهايد المعوض ليعطي قواعد شيف [VI]$_{a, b}$ [V]$_{a, b}$ومن تفاعل حامض الهايدرازايد مع أستيل أسيتون أو أثيل أسيتو أستيت نحصل على بايارازول$_{a,b}$[VII] و بايارازولين$_{a,b}$[VIII] على التوالي .شخصت جميع المركبات المحضرة بوساطة قياس درجات أنصهارها ,طيف FTIR و^{1}H NMR(لبعض منها)

الكلمات المفتاحية :الجالكون,قواعدشف ,الأيزوكزولين, البايارازول, البايارازولين