Iraqi Journal of Market Research and Consumer Protection
(2024) 16(1): 1-12.
DOI: http://dx.doi.org/10.28936/jmracpc16.1.2024.(1)
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المجلة العراقية لبحوث السوق
وحماية المستهلك

NEW METHDOLOGY OF SYNTHESIS PYRAZOLO- THIAZOLO DERIVATIVES WITH STUDY ANTIMICROBAL ACTIVITIES

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Received 9/2/ 2023, Accepted 25/ 6/ 2023, Published 30/ 6/ 2024

ABSTRACT

In this contribution, new derivatives of thiosemicarbazon, cyclic thiazolidinone, yilidine thiazolidinone, and parazolo-thiazole were synthesized, starting from the reaction of 2 -aminobenzothiazole with *p*-bromo phenacyl bromide to give the first step of 2(4-bromo phenyl) imidazole (2,1 -b) benzo thiazole (1). Compound (1) then was subjected under Viels Myer Haack to yield 3-carbaldehyde linking with imidazo-benzothiazol (2). Compound (2) condensed with thiosemicarbazide togivenewsynthesis of thiosemicarbazon derivative (3). Compound 4-Oxo-1,3-thiazolidine (4) was constituted from reacting compound (3) with ethyl chloroacetate, while Thiazolidinone derivative ((5 was formed by reacting compound (4) with aromatic aldehyde to give 4-Oxo-1,3-thiazolidine-5 benzylidene linking with imidazo-benzothiazol (5). Compound (5) was then reacted with hydrazine hydride to give fused ring of pyrazolo thaizolo derivatives (6). In the finally, azo methene groups were opening by two reagents are (acetic anhydride and 4-nitro benzoyl chloride) to give new derivatives of hydrazone contacted with imidazobenzothiazol (7) and (8) compound respectively. All the prepared compounds were identified by Fourier Transform Infrared (FT-IR), Proton nuclear magnetic resonance (^{1} H-NMR) and Carbon nuclear magnetic resonance (^{13}C-NMR) spectra. Some of the synthesized compounds were evaluated via biological activity.

Keywords: Imidazo (2,1 -b) benzothiazol , parazolo –thiazolo, Azomethene group, Antimicrobial Activities.

طريقه جديدة لتخليق مشتقات البايروزول - ثايازول مع دراسة نشاط مضادات الميكروبات

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الخلاصة

في هذه المساهمه، خلقت مركبات جديده من ثاياسيماكاربازون، حلقات الثايازوليدينات، اليلد ثايازوليدينات وحلقات ملتحمه من بايروزو- ثايازول من تفاعل المادة الأولية 2 –امينو بنزوثايازول مع برومو فنسيل بروماید لتشكل في الخطوه الاولى 2-(4- برومو فنيل) ايميدازو (2-1-b) بنزوثايازول (1). المركب الاول خضع لتفاعل فلز –مايرهاك ليعطي مجموعه الالديهايد في الموقع الثالث مركب (2)، وفي الخطوة التاليه تم تكاثف ثاياسيماكاربازيد مع المركب الثاني لتعطي مشتقات جديده من ثاياسيماكاربازون (3). مشتقات الثاياسيماكاربازون اغلقت بواسطه مركب اثيل كلورو اسيتات لتشكل حلقه الثايازوليدين (4).4-اوكسو (1و3)ثايازوليدين 5-بنزيلدين (5) تكون من تفاعل المركب (4)مع مختلف الالديهايدات الاروماتيه.بعد ذلك عومل المركب (5) مع هيدرازين هدراید ليكون حلقات ملتحمه من البايرازول – ثايازول (6). في الخطوات الاخيره لهذا العمل تم فتح مجموعه الايزوميثين وذلك من خلال معاملة مركب (6) مع الكواشف

*** The research is extracted from the doctoral thesis of the first researcher.**

(استيك انهدرايد، 4 نترو بنزويل كلورايد) لتعطي مشتقات جديده من مركبات الهيدرازينو(7)و (8)على التوالي . جميع المركبات تم فحصها بواسطه FT-IR وبعض منها تم فحصها بواسطه ^{1}H-NMRو C-NMR ^{13}،وكذلك تم دراسه مضاد نشاط الميكروبات لهذه المشتقات الجديده.

الكلمات المفتاحية: ايميدازو (1،2 – b) بنزوثايازول، بايرازول–ثايازول .فتح اصره الايزوميثين، مضادات الميكروبات.

INTRODUCTION

Heterocyclic compounds are important major objectives of organic synthesis **(Karam *et al.*, 2022)**, the imidazo benzo (2, 1 -b) thiazole compound is a heterocyclic compound including divers' significant biological activities **(Khalil& Khalal, 2021)** such as, anti-cancer, antimicrobial **(Al-Sultani & Al-Lami, 2021 .(**Aldehydes are one type carbonyl compounds are widly found in food products. The formation carbaldehyde belong to oxidation of fatty acid and higher alcohols, aldol condensation, Strecker degradation, and Villismyer haack reaction. The studies have proved the functional group of carbonyls have a very biological active such as antibacterial, antioxidant, anti-fungal, and some them used as an anti-and analge **(Al-Lami, 2015).**

Thiosemecarbazons are most of the intermediates compounds for the organic synthesis, the imine bond (-N=CH-) in this compounds are useful in particular for the preparation of many heterocyclic compounds, it has S/N regoselective nucleophile can able synthesis new substituent derivatives from ring closure of thiosemicarbazone such as indol, thiazol, thiadiazol, traizol ect. All this compound have wide biological activities **(Dawood *et al.*, 2010)**. Thiazolidinone heterocyclic compounds contained of five-member ring with with two heteroatoms sulpher and nitrogen**, (Ayyash *et al* 2019)** thiazolidinone compound has a wide of pharmaceutical significance in various drugs for examples antimycobacterial, antimicrobial, anticancer. Also, many clinically used drugs contain thiazolidinone ring in their skeletons such as antibiotic actithiazic acid **(Hussein & Al-Lami, 2022)** 4-Oxo-1,3-thiazolidine-5 benzylidene carrying functional group as hydroxy, methoxy, nitro and chloro groups ect, linking with imidazo benzo (2,1 -b) thiazole derivatives were synthesized from thiazolidine and aromatic aldehyde, these compounds have biological antimicrobial activity against gram positive and gram negative bacteria, yeasts and mould **(Turgut *et al* .,2007)**. Parazolo-thiazolo is a fused Five-Five membered rings system without ring junction heteroatoms, More specifically, these ring systems have two heteroatoms of nitrogen in the five-membered ring and other fused ring has one heteroatom of nitrogen and sulpher **(Kasralikar *et al.*, 2019)**. The main types of fused pyrazolo thiazole showed in (Figure, 1).

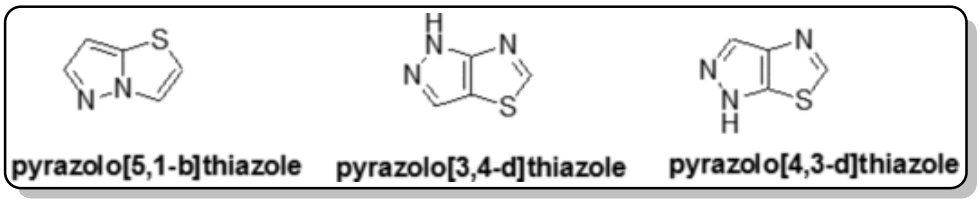


Fig (1): Types of pyrazolo thiazole.

These compounds have a wide spectrum of bioactivity such as antioxidants and anti-inflammatory agents. Some of them exhibit appreciable activity as virucides, bacteriocides, cytokine inducers, and immunomodulators. **(Kasralikar *et al.*, 2019)**. The studied proved a number of hydrazide compounds have been reported for their medical applications, hydrazide derivatives are present in many bioactive molecules and display a wide variety of biological

activities, such as antibacterial, antitubercular, antifungal, anticancer, anti-inflammatory, anticonvulsant, antiviral, and antiprotozoal action P. **(Preethi Kumari *et al* ., 2017).**

MATERIALS AND METHODS

Material and instrumentation

Corporations were supplied from diverse all chemicals such as Thomas baker, Merck, BDH, Sigma-Aldrich. End of reaction of all compounds were checked on aluminum –coated TLC plates 60 F245[E. MERCK] by using ethyl acetate and Petroleum ether and imagined under iodine vapor.

Melting points were determined on an electro thermal melting point (Stuart Germany), and they were uncorrected. Infrared spectra resolves were done as a KBR disk in range of (400-4000 cm^{-1}) FT-IR Shimadzu was used to record at university of Bagdad /College of science. The proton ^{1}H-NMR and ^{13}C-NMR spectra operating at 400 MH_Z and 100 MH_Z respectively in DMSO-d_6, measurements are performed at Collage Sharif University of Technology /Tehran/ Iran.

Preparation of 2(4-bromo phenyl) imidazo benzo [2,1 -b] thiazol (1) (Al-Sultani *et al.*, 2021)

Equimolar quantity (0.01 mol,3.2 gm) of 2-amino-benzothiazole, 4-bromophenyl phencyl bromide respectively were dissolved in abs. ethanol (40ml). The mixture was refluxed for 6 h. The solid compound was filtered and recrystallized by suitable solvent. All physical properties of compound **(1)** represent with molecular formula: $C_{14}H_9N_2S$ Br, Color: Off white, Yield :85%, M.P160: Re-crystallization solvent: Absolut ethanol.

Preparation of 2-(4-bromophenyl) imidazo (2, 1-b) benzothiazole-3-carbaldyhade (2) (Al-Lami, 2015; Alyaa, 2020).

2-aminobenzothiazol (0.01 mol, 1.5 gm) dissolved in(15mL) of DMF with kept on temperature (0-5) ^{0}C, then added drops wise (1mL) phosphorous oxychloride ($POCl_3$) with stirring then refluxed about 5 h. After that the obtain solid compound was filtered and purified from ethanol. All physical properties to compound **(2)** represented with molecular formula: $C_{16}H_9N_2OBr$, Color: White, Yield :75%, M.P: 256 C^0, Re-crystallization solvent: Ethyl acetate.

Synthesis of 2(4-bromophenyl) imidazo (2, 1-b) benzothiazole-3-thiosemicarbazone (3) (AL-Khazraji& Ahmed, 2022; Mousa& Jassim, 2021).

an equimolar of aldehyde (0.01 mol, 1gm) and thiosimecarbazide (0.01 mol ,3.12gm) in absolute ethanol (20 mL) with 2-3 drops of glacial acetic acid, were refluxed for 5 h. The mixture was cooled to room temperature, and the solid product was washed with cold water, purified with ethanol to yield compounds(3). all physical properties to compound represented with molecular formula: $C_{16}H_{12}N_5OSBr$, Color: Yellow, Yield :70%, M.P: 195-197 ^{0}C, Re-crystallization solvent: Ethyl acetate

Synthesis of 2(4-bromopheny) limidazo (2, 1-b) benzothiazole-3-yl) methylenehydrazineyl-thiazol-4(5H)-one (4) (Khalil & Khalal, 2021).

A mixture of thiosemicarbazone (0.01mol,1.2 gm) with (0.01mol,0.5 mL) of ethyl chloroacetate and sodium acetate (0.04 mol) was also added in absolute ethanol (20mL) and the resulting mixture was refluxed for 7 h. The reaction monitored by TLC. The crude product was cooled at room temperature, then poured onto ice-water. The resulted precipitate was filtered, washed with water and dried.

Syntheses of 2(4-bromophenyl) imidazo (2,1-b) benzothiazole-(3- methylene hydrazineyl-(4-Oxo-1,3-thiazolidine)-5 benzylidene (5) (Kasralikar *et al.*, 2019)

Compound (4) (0.01 mol, 0.5gm) was mixed with (0.01mol, 0.3 gm) of aromatic aldehyde (*p*-Nitro benzaldehyde, O-hydroxy benzaldehyde with anhydrous sodium acetate (0.015 mol) dissolved in (10 mL) of glacial acetic acid. The mixture was heated under reflux for 7 h. The reaction was monitored by TLC. The crude product was cooled to room temperature, the resulted precipitate was filtered, washed with water, and dried to give compound (5). All physical properties of compound **(5)** are listed in (Table, 1).

Table (1): The physical properties of the compound [5P-5Q].

Comp. NO	M.F	Color	Yield (%)	M.P	Re- cryst. solvent
5P	$C_{23}H_{17}N_6SO_3$ Br	yellow	85	279	Chloroform
5Q	$C_{23}H_{18}N_5O_2S$ Br	yellow	75	330	ethanol

Synthesis of of 2(4-bromophenyl) imidazo (2, 1-b) benzothiazole-3- methylene hydrazineyl - [3-benzyl -5H-pyrazolo-[3,4-d]-1,3-thiazole] (6 P-6Q) (El-Hamouly *et al*., 2011).

A mixture of compound (5) -(4-Oxo-1,3-thiazolidine)-5 benzylidene (0.02 mol, 1gm) and hydrazine hydride (0.06 mol, 1mL) was refluxed in ethanol (20 mL) in the presence of few drops of hydrochloric acid for 5 hrs. The crude product was cooled to room temperature, the resulted precipitate was filtered, washed with water, dried. (Table 2) explained all physical properties to compound (6).

Table (2): The physical properties of the compound [6P-6Q].

Comp. No	M.F	Color	Yield(%)	M.P	Re-cryst.solvent
6P	$C_{23}H_{19}N_8O_2S$ Br	yellow	75	270	Chloroform
6Q	$C_{23}H_{20}N_7OS$ Br	yellow	85	380	ethanol

Synthesis of 2(4-bromophenyl) imidazo (2, 1-b) benzothiazole-3- methyl acetate hydrazono-1-acytel [3-benzyl -5H-pyrazolo-[3,4-d]-1,3-thiazole] (7P-7Q) (Zainab, 2008)

(0.5 gm,0.005 mol) of Schiff bases dissolved in (15 mL) of dry benzene then added (0.51 gm ,0.005 mol) from acetic anhydride dissolved in (10 mL) from benzene after that

refluxed about 2h. After that the obtain solid compound was filtered and purified from suitable solvent. (Table 3) explained all physical properties to compound (23).

Table (3): The physical properties of the compound [7P-7Q].

Comp.NO	M.F	Color	Yield (%)	M.P	Re-cryst.solvent
7P	$C_{27}H_{25}N_8O_5S$ Br	Brown	75	279	Chloroform
7Q	$C_{27}H_{26}N_7O_4S$ Br	yellow	75	280	ethanol

Synthesis of 2(4-bromophenyl) imidazo (2, 1-b) benzothiazole-3- chloro methyl -4-nitro benzo hydrazino [3-benzyl -5H-pyrazolo-[3,4-d]-1,3-thiazole](8P-8Q) (Zainab ,2008)

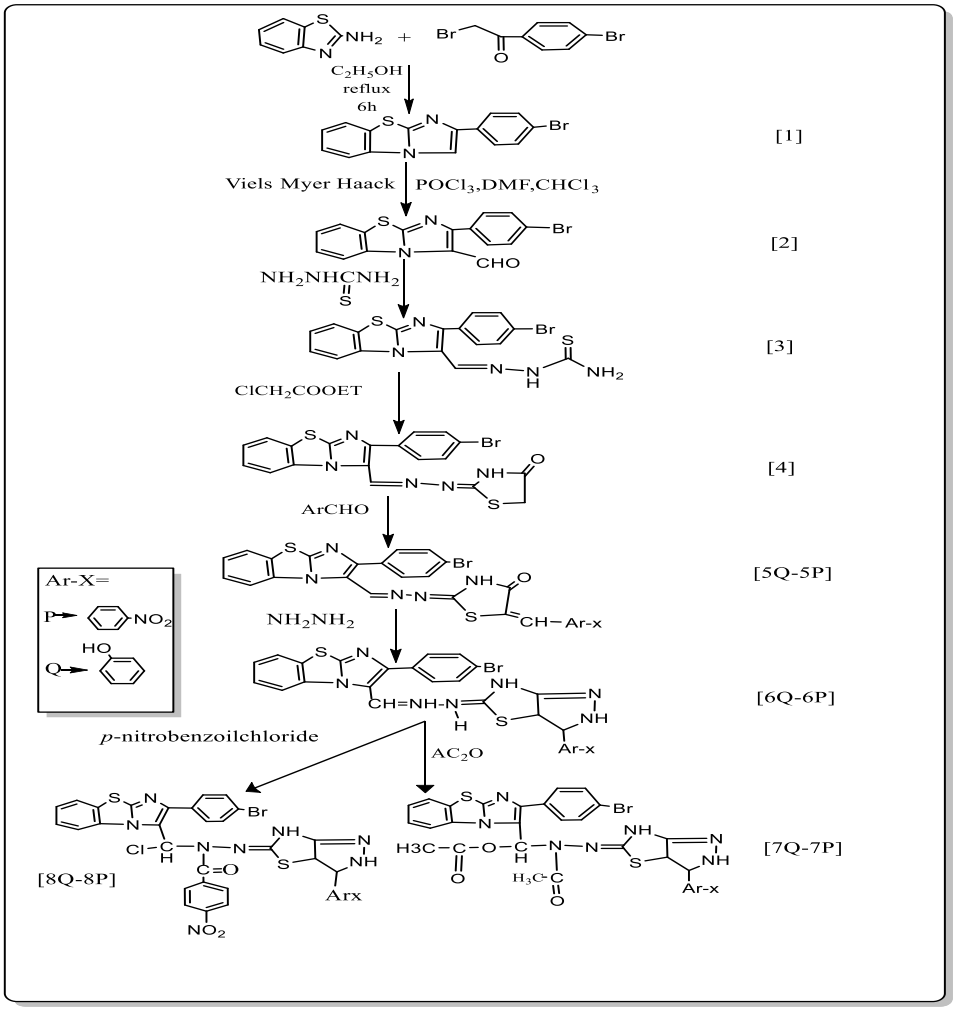
Schiff bases (0.005 mol, 0.5 gm) dissolved in (15 mL) of dry benzene then added (0.005 mol ,0.51 gm) from 4-nitro benzoyl chloride dissolved in (10 mL) from benzene after that refluxed about 3 hrs. After that the obtain solid compound was filtered and purified from suitable solvent. (Table 4) explained all physical properties to compound (24).

Table (4): The physical properties of the compound [8P-8Q].

Comp.NO	M.F	Color	Yield (%)	M.P	Re-cryst.solvent
8P	$C_{31}H_{26}N_{11}O_5S$ BrCl	yellow	75	285	Chloroform
8Q	$C_{31}H_{27}N_{10}O_4S$ BrCl	yellow	89	288	ethanol

RESULTS AND DISCUSSION

All the reactions are explained in Scheme 1



Scheme (1): Synthesis steps of compounds.

In this work, the imidazo benzo (2,1 –b) thiazole compound **(1)** was obtained from the reaction of 2- amino benzo thiazole with 4-(2- bromo phenacyl bromide) and characterization by FTIR spectrum showed bands at 1645 due to ν C=N imidazo ring, ν C=C aromatic at1575, other bands showed in(Table5)

Table (5): Characteristic absorption bands in FT-IR spectra of compound (1) in Cm^{-1}

Comp No.	ν(C-H) Aromatic	νC=N imidazo ring	ν C=C Arom.	Other bands
1	3051	1645	1575 1490	ν(C-N) 1336 ν(C-C)937 ν(C-Br)744

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compound (1) reacted with thiosemicarbazone to give formal derivatives **(2)** was showed absorption band at 1685 cm^{-1} due to ν C=O, at (1591) cm^{-1}due to ν C=N, other bands showed in (Table 6).

Table (6): Characteristic absorption bands in FT-IR spectra of compound (2)in Cm^{-1}

Comp No.	ν(C-H) Aromatic	ν C=O	νC=N imidazoring	ν C=C Arom.	Other bands
2	3076	1685	1591	1556 1433	ν(C-N) 1321 ν(C-C)927 ν(C-Br)781

Thiosemicarbazon compounds **(3)** was prepared by reacted formal compound with thiosemicarbazide were subjected to diagnosis by FT-IR was showed absorption band at (3367-3498) cm^{-1} belong to ν NH_2, (CH=N) at 1662cm^{-1}, ν (C=N) at1640 cm^{-1}.other bands in (Table 7).

Table (7): Characteristic absorption bands in FT-IR spectra of compound (3)in Cm^{-1}

Comp No.	ν NH_2	ν NH	ν CH=N	νC=N imidazo ring	ν C=C Arom.	Other bands
3	3367-3498	3161	1662	1640	1573 1490	ν (CH)aldehyde 1725 ν (CH)Arom3004 ν(C-N) 1323 ν(C-C)925 ν(C-Br)748

In **^{1}H-NMR** compound **(3)** was result as fellow: at 4.49 ppm (s,2H,NH_2), at (7.20-8.56)ppm, (m,8H,ArH), at (8.66)ppm (s,1H,CH=N), at (9.07)ppm (s,1H,NHC=S).

Thiosemecarbazone was reacted with ethylchloroacetate and anhydrous sodium acetate in ethanol to give five member ring is thiazolidinone ring compound **(4)** confirmed by the FT-IR, showed the disappearances of NH_2 group bands of thiosemicarbazons group and the appearances of new characteristic bands at (1708cm^{-1}) belong to the stretching vibration of (C=O) of lactam groups. All FT-IR spectra of compounds **(4)** are listed in (Table, 8).

Table (8): Characteristic absorption bands in FT-IR spectra of compound (4) in Cm^{-1}

Comp No.	ν NH	ν C=O Lactam	ν CH=N	ν C=N	ν C=C Arom	Other bands
4	3101	1708	1662	1652 1595	1595 1440	(CH)Arom.3076 ν(C-N) 1396 ν(C-C)939 ν(C-Br)748

Compound **(4)** Characteristic by ^{1}**H-NMR** the result as fellow: at (4.02-4.07) ppm (s,2H,CH_2), at (7.37 -8.48) ppm (m,8H,ArH), at (8.53)ppm (s,1H,CH=N), at (9.69)ppm (s,1H,NHC=S)

Thiozolidinone compound reacted with different aromatic aldehyde to give compound **(5P-5Q),** showed stretch band at1697-1699 cm^{-1} belong to (C=O),at 1604-1614 cm^{-1}due to (C=C)alkene,at1652-1666 cm^{-1} belong to ν (CH=N), other bands showed in(Table 9),

Table (9): Characteristic absorption bands in FT-IR spectra of compound (5P- 5Q)in Cm^{-1}

Comp No.	**ν NH**	**ν C=O**	**ν CH=N**	**ν C=C Alkene**	**ν C=N**	**Other bands**
5P	3191	1699	1652	1604	1564	CH Arom. 3004 ν(NO_2)Asym. 1512,Sym 1350, ν(C=C)Arom. 1463,1550 ν(C-N) 1319, ν(C-C)927, ν(C-S) 819, ν(C-Br)769
5Q	3137	1697	1666	1614	1573	ν(O-H)3434, ν(C=C)Arom. 1541,1440 ν(C-N) 1317, ν(C-C)925, ν(C-S) 788, ν(C-Br)748

Compound (5P) conformed by ^{1}H-NMR,^{13}C-NMR spectra all data appeared in (Table 10).

Table (10): Characteristic absorption bands in ^{1}H-NMR α^{13}CNMRspectra of compound(5P)

Comp.	**^{1}H-NMR**	**^{13}CNMR**
5P	at (7.38) ppm (s,1H,C=CH), at(7.40-8.42)ppm(m,8H,ArH), at (8.50) ppm(s,1H, CH=N) ,at(9.26)ppm (s,1H,NHC=S).	(21.54) ppm(C-NO_2) , (116.47-133.79) ppm(C=C) arom, at (149.01) ppm(=CH), at (150.76) ppm (CH=N), at (178.21) ppm carbonyl group

Compound (5) cyclic by hydrazine hydride to give pyrazolo –thiazolo derivation **(6Q-6P)** an characterization by FTIR and results as fellow: compound **(6p)** appeared band absorption at 1614-1600 cm^{-1} due to (C=N) pyrazolo- thiazolo ring ,1649-1662 cm^{-1} due to CH=N . all FT-IR spectra of compounds (6) are listed in (Table, 11).

Table (11): Characteristic absorption bandsin FT-IR spectra of compound(6p-6Q)inCm^{-1}

Comp No.	ν NH	ν CH=N	ν C=N pyrazo-thiazol and imidazo ring	ν C=C Arom	Other bands
6P	3149	1629	1595 1564	1550 1490	CH Ar.3070 ν(NO_2)Asym1531,Sym 1394, ν(C-N) 1317, ν(C-C)929, ν(C-S) 802, ν(C-Br)746
6Q	3137	1623	1600 1564	1598 1488	ν(O-H)3448, (CH)Arom 3074, ν(C-N) 1319, ν(C-C)931, ν(C-S) 802, ν(C-Br)748

Compound**(6P)** confirmed by **^{1}H-NMR**and results as fellow at (7.388.35) ppm (m,8H, ArH), at(8.55)ppm(s,1H,CH=N),at(9.60)ppm (s,1H,NHC=S). In compound (7) the action mechanism **(Zainab, 2008**) was Azomethen group opening by reagent (acetic anhydride) .see bellow to Scheme (2).

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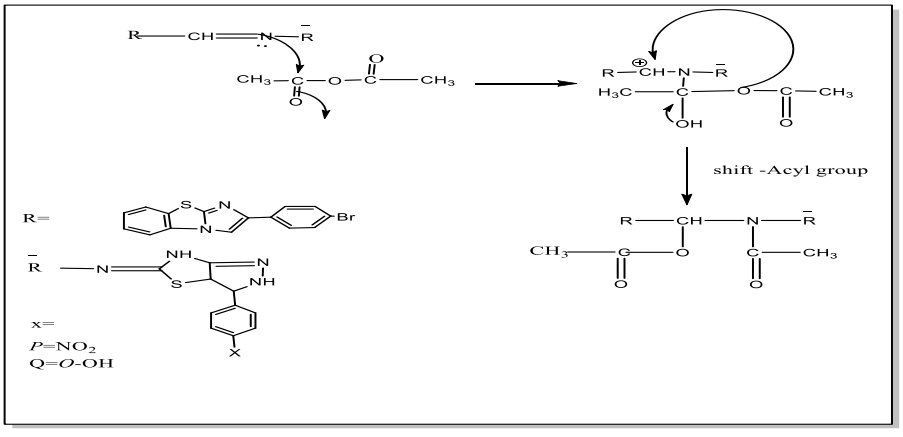

Scheme (2): Mechanism of compound (7).

Compound (7P-7Q) in FTIR spectra showed disappearances stretch bands belong Azomethen and appearance carbonyl lactams at (1685-1703) cm^{-1}, and carbonyl ester at (1718-1720). All FT-IR spectra of compounds (7P-7Q) appear in (Table12)

Table (12): Characteristic absorption bands in HNMR spectra of compound (7P-7Q)

Comp No.	ν NH	ν C=O Ester, Amide	ν C=N pyrazo thiazol,and imidazo ring	ν C=C Arom	Other bands
7P	3139	1718 1703	1627 1595	1490	ν CH Ar3011 ν(NO2)Asym 1531,Sym 1394, ν(C-N) 1317, ν(C-C)927, ν(C-S) 802, ν(C-Br)746
7Q	3228	1720 1685	1623 1575	1535	ν(O-H)3425 (CH)Ar.3028, ν(C-N) 1363, ν(C-C)925, ν(C-S) 802, ν(C-Br)750

Compound **7P** confirmed by **^{1}H-NMR** and results as fellow: at (2.27) ppm(s,3H,CH_3 lactam), At (2.27) ppm(s,3H,CH_3 lactone), at (7.43-8.35)ppm (m,8H,Ar-H) ,at(9.70)ppm (s,1H,NHC=S) Azomethen opening by reagent(4-nitro benzoyl chloride) to give (8P-8Q)compound, the FTIR spectra showed disappearances stretch bands belong to Azomethen group and appearance carbonyl lactam at (1693-1695) cm^{-1}.All FT-IR spectra of compounds(8P-8Q) appeared in (Table, 13)

Table (13): Characteristic absorption bands in HNMR spectra of compound (8P-8Q)

Comp No.	**ν NH**	**ν C=O Amide**	**ν C=N pyrazo thiazol,and imidazo ring**	**ν C=C Arom**	**Other bands.**
8P	3114	1693	1604 1595	1550 1492	ν CH Ar3062 ν(NO_2)Asym 1525,Sym 1348, ν(C-N) 1313, ν(C-C)931, ν(C-S) 800, ν(C-Br)717
8Q	3261	1695	1649 1573		ν(O-H)3438 (CH)Ar.3028, ν(NO_2)Asym 1502,Sym 1373 ν(C-N) 1363, ν(C-C)925, ν(C-S) 786, ν(C-Br)767

Biological Activities (Alyaa,2020)

In this work synthesized compounds have active moieties in their structures, therefor these compounds to possess a wide spectrum from biological activity. Some of the prepared compounds were tested against two type of bacteria (Staphylococcus aureus (+*ve*), and Escherichia coli(-*ve*), and two type of anti-fungal (Candia albicans, Asp. niger). As shown (Table 14).

Table (14): Anti-bacterial activity for some prepared compounds

Comp. NO.	Staphylococcus Aureus(+ve)	Escherichia coli (-ve)	Candia albicans	Asp.niger
5p	25mm	--	16mm	22mm
5Q	-	-	23mm	22mm
8p	14mm	25mm	-	-
8Q	-	25mm	23mm	-

CONCLUSIONS

From this work, we have successfully prepared new heterocyclic compound as (thiazolidinone and parazolo- thiazolo) rings by using start martials 2- amino benzothiazol ,fused ring have very important biological activity, all new derivations analytical and spectral data FT-IR and some of them analytical by (^{1}HNMR,^{13}CNMR)proved the proposed structures.

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