

Preparation Some New Heterocyclic Compounds Derived from Schiff Bases and Evaluation its Biological Activity

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Abstract: The research include preparation some new 2, 3-dihydroquinazolin-4 (1H) -one and oxazepine derivatives. Schiff bases derivatives (1-5) formation from condensation 4-amino-N- (p-tolylcarbamothioyl) benzamide with some aromatic aldehyde (p-Bromobenzaldehyde; p-Nitrobenzaldehyde; p-Chloro benzaldehyde; 2, 4-di hydroxybenzaldehyde and 2, 4-di methoxybenzaldehyde). The solvent is ethanol and in the presence of glacial acetic acid. Then two chemical reagents (anthranilic acid and maleic anhydride) used to cyclization of the prepared Schiff bases to form 2, 3-dihydroquinazolin-4 (1H) -one (6-10), oxazepine (11-15) derivatives respectively. The chemical structure of new synthesized compounds characterized (infrared and some derivatives by Nuclear Magnetic Resonance (¹HNMR, ¹³C-NMR) and measurements some of its physical features and some specific reactions. and checked through T.L.C. Also, the biological activity of some compounds was tested against bacteria.

Keywords: Schiff Bases; 2, 3-Dihydroquinazolin-4 (1H) -one; Oxazepine; Antibacterial.

1. Introduction

Heterocyclic Compounds containing an azomethine group (-CH=N-), called as Schiff bases. The first synthesis of imine was done by Hugo Schiff in 1864[1]. The Schiff base is formed by react compounds having active carbonyl groups with primary amines in presence acid (2). Schiff bases possess a wide variety of biological activities such as antimicrobial activity (3), antileishmanial (4), anti-inflammatory (5), anti HIV (6), Anticonvulsant (7), anticancer (8), antifungal (9) and antiproliferative (10). Schiff bases derivatives are important in medical field. Quinazolinone are an important class of heterocyclic compounds were prepared by reaction Schiff bases with anthranilic acid. 2, 3-Dihydro-4 (1H) -quinazolinones and its derivatives are very interesting in pharmacological and biological activities such as anticonvulsant and antitumor hypnotic, analgesic, antitussive, antibacterial, antifungal activity, diuretic, anti-inflammatory anti-hypertensive activities (11-12). Oxazepine is seven member ring containing two hetero atoms (oxygen and nitrogen) (13). The oxazepine can be prepared by the cycloaddition of schiff bases with anhydride (14). Oxazepine and its derivatives have very important biological activities such as analgesic, enzyme inhibitors, Amoxapine, antidepressant and psychoactive drugs (15-17). A part of our research, we want to prepare many derivatives of (2, 3-Dihydroquinazolinone and Oxazepine) and investigate its antimicrobial activities.

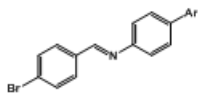
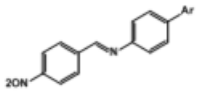
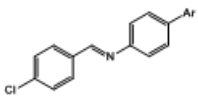
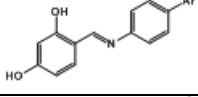
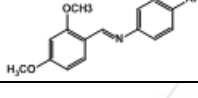
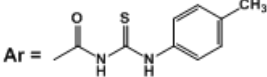
2. Experimental Part

- 1) Melting points were recorded using digital Stuart scientific SMP3 melting point apparatus and are uncorrected.
- 2) FTIR spectra were recorded on SHIMAZU FTIR-8400 using KBr discs in the (4000-600) cm⁻¹ spectral range.
- 3) ¹HNMR and ¹³CNMR were recorded on Burker 500MHz instrument using CDCl₃-d as solvent and TMS as internal reference.
- 4) Thin layer chromatography (TLC) was carried out using Fertigfollenpre coated sheets type polygram silica and the plates were developed with iodine vapor.

Preparation of (E) -4-N- (p-tolylcarbamothioyl) acetamide-N- (4-substitutedbenzylidene) aniline (1-3) and (E) -4-N- (p-tolylcarbamothioyl) acetamide-N- (2, 4-di substitutedbenzylidene) aniline (4-5) (18)

Schiff bases derivatives prepared by reaction (p-Bromobenzaldehyde; p-Nitrobenzaldehyde; p-Chloro benzaldehyde; 2, 4-di hydroxybenzaldehyde and 2, 4-di methoxybenzaldehyde) (0.01 mol.) with N-[(4-aminophenyl) carbamothioyl] benzamide (0.01 mol.) (19) in ethanol for (3-7 hour) with (1-2 drops) of glacial CH₃COOH. A precipitate was formed which collected by filtration and recrystallized by diethyl ether. The Physical features and the Fourier transform infrared values for the compounds in Table (1).

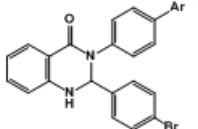
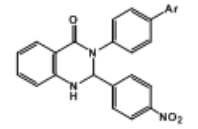
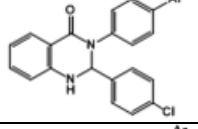
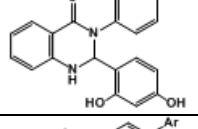
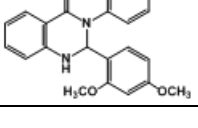
Table 1: The Physical features and the Fourier transform infrared values for derivatives (1-5).

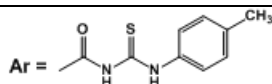
Comp.No.	Physical features				Fourier Transform Infrared cm^{-1}					
	Chemical Structures	Melting Point $^{\circ}\text{C}$	Yield %	Colors	$\nu\text{N-H}$	$\nu\text{C-H arom.}$	$\nu\text{C=N}$	$\nu\text{C=S}$	$\nu\text{C=O}$	Other important bands
1		167-169	80	brown	3180	3041	1668	1259	1668	$\nu\text{C-Br}$ 977 vp-position 825
2		177-179	90	pale yellow	3185	3041	1668	1259	1649	NO_2 Asym.1515 sym.1344 vpara-position 835
3		175-178	80	Off-white	3290	3031	1649	1244	1670	$\nu\text{C-Cl}$ 1093 vp-position 819
4		183-184	75	Gray	3270	3031	1670	1259	1650	$\nu\text{O-H}$ 3404
5		184-186	85	brown	3232	3051	1668	1263	1670	$\nu\text{C-O-C}$ 1112
										

Preparation of 3- (4- N- (p-tolylcarbamothioyl) acetamide) -2- (4- substituted phenyl) -2, 3-dihydroquinazolin-4 (1H) -one (6-8) and 3- (4- N- (p-tolylcarbamothioyl) acetamide) -2- (2, 4- substituted phenyl) -2, 3-dihydroquinazolin-4 (1H) -one (9-10) (20).

A solution of 2- amino benzoic acid (anthranilic acid) (0.02mol) and Schiff base (1-5) in dioxane was added. The solution was heated under reflux temperature for 18hrs. after that sidue was treated with 10% of sodium bicarbonate. Then filtered and recrystallized by ethanol. The Physical features and the Fourier transform infrared values for the compounds in Table (2).

Table 2: The Physical features and the Fourier transform infrared values for derivatives (6-10).

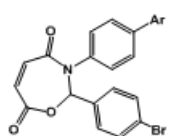
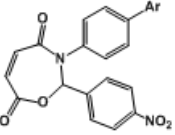
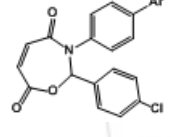
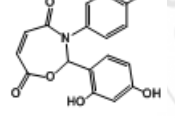
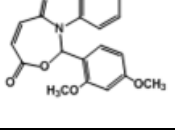
Comp. No.	Physical Features				Fourier Transform Infrared cm^{-1}				
	Chemical Structures	Melting Point $^{\circ}\text{C}$	Yield %	Colors	$\nu\text{N-H}$	$\nu\text{C-H arom.}$	$\nu\text{C=O}$	$\nu\text{C=S}$	Other important bands
6		192-194	65	white	3331 3277	3050	1676 1655	1250	$\nu\text{C-Br}$ 882 vp-position 830 $\nu\text{C-N}$ 1270
7		144-147	70	yellow	3342 3240	3050	1690 1658	1242	νNO_2 asym 1514 sym 1344 $\nu\text{C-N}$ 1319
8		182-184	70	Off-white	3338 3242	3070	1680 1660	1244	ν (C-Cl) 1088 vp-position 835 $\nu\text{C-N}$ 1244
9		210decomp.	60	brown	3340 3280	3040	1683 1640	1261	$\nu\text{O-H}$ 3420 $\nu\text{C-N}$ 1261
10		217decomp.	75	white	3444 3259	3031	1681 1651	1247	ν (C-O-C) 1152 $\nu\text{C-N}$ 1247

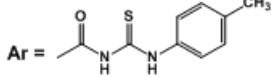


Preparation of 3- (4-N- (p-tolylcarbamothioyl) acetamidephenyl) -2- (4-substituted phenyl) -2, 3-dihydro-1, 3-oxazepine-4, 7-dione (11-13) and 3- (4-N- (p-tolylcarbamothioyl) acetamidephenyl) -2- (2, 4-substituted phenyl) -2, 3-dihydro-1, 3-oxazepine-4, 7-dione (14-15) (21).

Schiff base (1-5) (0.01mol) in 10 ml of dry benzene and (0.01mol) of maleic anhydride dissolved in 10ml of dry benzene and refluxed in a water bath for (6-8) hrs. Then filtered and recrystallized by ethanol. The Physical features and the Fourier transform infrared values for the compounds in Table (3).

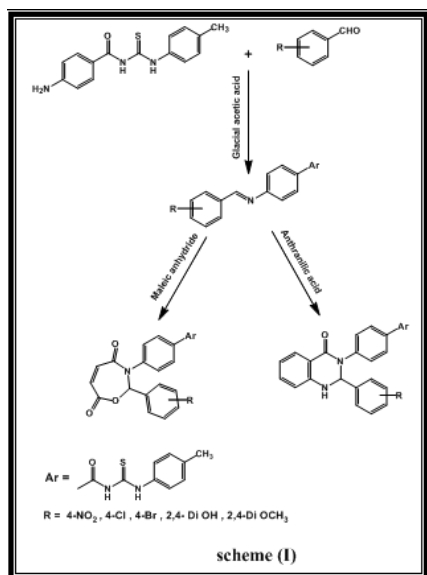
Table 3: The Physical features and the Fourier transform infrared values for derivatives (11-15)

Comp.No.	Physical features				Fourier Transform Infrared cm^{-1}				
	Chemical Structures.	Melting Point $^{\circ}\text{C}$	Yield %	Color.	$\nu\text{N-H}$.	$\nu\text{C-H}$. arom.	$\nu\text{C=O}$.	$\nu\text{C=S}$.	Other Important Bands
11		222decomp.	70	Light brown	3230	3066	1650	1255	ν (C=O) maleic 1780 $\nu\text{C-Br}$ 898 vp-position 821 $\nu\text{C-N}$ 1178 $\nu\text{C-O}$ 1255
12		169-172	88	brown	3261	3070	1655	1253	ν (C=O) maleic 1710 νNO_2 asym 1517 sym 1342 $\nu\text{C-N}$ 1178 $\nu\text{C-O}$ 1253
13		199-201	75	Pale-yellow	3220	3066	1674	1251	ν (C=O) maleic 1710 ν (C-Cl) 1093 vp-position 769 $\nu\text{C-N}$ 1176 $\nu\text{C-O}$ 1251
14		155-158	65	brown	3185	3029	1600	1257	ν (C=O) maleic 1780 $\nu\text{O-H}$ 3460 $\nu\text{C-N}$ 1176 $\nu\text{C-O}$ 1257
15		170-173	65	deep brown	3163	3060	1676	1265	ν (C=O) maleic 1710 ν (C-O-C) 1140 $\nu\text{C-N}$ 1170 $\nu\text{C-O}$ 1270

Ar = 

3. Results and Discussion

Preparation of new 2, 3-Dihydroquinazolinone and Oxazepine derivatives found in scheme (I).



The new azomethine derivatives (Schiff bases) (1-5) were synthesis by condensation of 4-amino-N-(p-tolylcarbamothioyl) benzamide with some substituted aromatic aldehydes in glacial acetic acid, the chemical structure for prepared compounds and Physical features in Table (1). The Fourier transform infrared values showed disappearance of absorption band for amine group and (C=N) band appearance at (1670-1649) cm^{-1} . Table (1) show other absorption bands for the substituted groups (22). Nuclear Magnetic Resonance spectrum ($^1\text{H-NMR}$) for compound (2) appear signals at ν 2.34 ppm (S, 3H, $-\text{CH}_3$); ν 6.40-8.33 ppm (m, 13H, Ar-H); ν 8.66 ppm (s, 1H, $-\text{CH}=\text{N}-$); ν 9.13 ppm (s, 1H, $-\text{Ph}-\text{NH}-\text{CS}$); and ν 10.33 ppm (s, 1H, $-\text{CS}-\text{NH}-\text{CO}-$). $^{13}\text{C-NMR}$ spectrum for this compound show signals in Table (5). The Schiff bases derivatives were cyclized by using two different reagent.

2, 3-Dihydroquinazolinone derivatives were prepared by reaction anthranilic acid (O-amino benzoic acid) with Schiff base in dioxane as a solvent. The Fourier transform infrared values show the appearance of NH vibration in (3331-3444) cm^{-1} and disappearance of absorption band for (C=N) at (1649-1670) cm^{-1} . The physical properties and FT-IR spectral data for compounds (6-10) in table (2). Nuclear Magnetic Resonance spectrum ($^1\text{H-NMR}$) spectrum for compound (5) appear signals ν 2.30 ppm (S, 3H, $-\text{CH}_3$); ν 5.30 ppm (s, 1H, $-\text{NH}-\text{C}$); ν 6.3 ppm (s, 1H, $-\text{N}-\text{CH}$); ν 6.30-8.46 ppm (m, 16H, Ph-H); ν 9.13 ppm (s, 1H, $-\text{Ph}-\text{NH}-\text{CS}$); ν 10.30 ppm (s, 1H, $-\text{CS}-\text{NH}-\text{CO}-$). $^{13}\text{C-NMR}$ spectrum for this compound show signals listed in Table (5). Nuclear Magnetic Resonance spectrum ($^1\text{H-NMR}$ and $^{13}\text{C-NMR}$) spectrum for comp. (10) show in table (4) and (5) respectively.

oxazepin derivatives (11-15) were prepared by cyclization azomethine derivatives (Schiff bases) (11-5) with maleic anhydride to result rings (seven membered) by cyclo addition reaction is classified as a [5+2] implying 5-atom component plus 2-atom component leading to 7-membered cyclic ring. The Physical features of oxazepin derivatives (11-15) in Table (3). The Fourier transform infrared values for the compounds (11-15) showed bands at (1780-1710) cm^{-1} due to the cyclic (C=O) stretching of ketone imide ring, the other substituted groups are listed in Table (5). $^1\text{H-NMR}$ spectral data of compounds (12 and 15) shows appearance signal for proton oxazepin ring and the same time disappearance of proton of azomethane group, the results listed in Table (4) and $^{13}\text{C-NMR}$ spectral data of compounds (12 and 15) show results listed in Table (5) (23).

Table 4: $^1\text{H-NMR}$ spectral data for some of the prepared compounds

Compound No.	$^1\text{H-NMR}$ data (ppm)
2	2.34 (S, 3H, $-\text{CH}_3$); 6.40-8.33 (m, 13H, ph-H); 8.66 (s, 1H, $-\text{CH}=\text{N}-$); 9.13 (s, 1H, $-\text{Ph}-\text{NH}-\text{CS}$); 10.33 (s, 1H, $-\text{CS}-\text{NH}-\text{CO}-$)
5	2.30 (S, 3H, $-\text{CH}_3$); 3.87 (S, 6H-OCH ₃); 7.63-8.80 (m, 11H, ph-H); 9.10 (s, 1H, $-\text{Ph}-\text{NH}-\text{CS}$); 10.22 (s, 1H, $-\text{CS}-\text{NH}-\text{CO}-$)
7	2.33 (S, 3H, $-\text{CH}_3$); 5.30 (s, 1H, $-\text{NH}-\text{C}$); 6.3 (s, 1H, $-\text{N}-\text{CH}$); 7.35-7.46 (m, 16H, ph-H); 9.13 (s, 1H, $-\text{Ph}-\text{NH}-\text{CS}$); 10.30 (s, 1H, $-\text{CS}-\text{NH}-\text{CO}-$)
10	2.35 (S, 3H, $-\text{CH}_3$); 3.84 (S, 6H-OCH ₃); 5.27 (s, 1H, $-\text{NH}-\text{C}$); 6.99-9.40 (m, 15H, ph-H); 9.10 (s, 1H, $-\text{CS}-\text{NH}-\text{Ph}$); 10.12 (s, 1H, $-\text{CO}-\text{NH}-\text{CS}-$)
12	2.40 (S, 3H, $-\text{CH}_3$); 7.30 (s, 1H, $-\text{CH}-\text{O}-\text{CO}$); 6.33-7.40 (m, 14H, ph-H); 9.28 (s, 1H, $-\text{Ph}-\text{NH}-\text{CS}$); 10.45 (s, 1H, $-\text{CS}-\text{NH}-\text{CO}-$)
15	2.33 (S, 3H, $-\text{CH}_3$); 3.80 (S, 6H-OCH ₃); 7.15 (s, 1H, $-\text{CH}-\text{O}-\text{CO}$); 6.30-7.42 (m, 17H, ph-H); 9.15 (s, 1H, $-\text{Ph}-\text{NH}-\text{CS}$); 10.22 (s, 1H, $-\text{CS}-\text{NH}-\text{CO}-$)

Table 5: $^{13}\text{C-NMR}$ spectral data for some of the prepared compounds

Compound No.	$^{13}\text{C-NMR}$ data (ppm)
2	21.2 (CH_3); 124.0-137.2 (C- arom. rings); 156.55 ($-\text{C}=\text{N}-$); 167.33 (C=O); 179.30 ($-\text{C}=\text{S}$)
5	21.4 (CH_3); 55.6 (OCH ₃); 122.2-139.33 (C- arom. rings); 166.6 (C=O); 180.34 (C=S)
7	21.4 (CH_3); 122.0-135.6 (C- aromatic rings); 165.33 (C=O); 181.0 (C=S)
10	21.3 (CH_3); 56.6 (OCH ₃); 126.0-137.2 (C- aromatic rings); 161.1 (C=O); 181.3 (C=S)
12	21.1 (CH_3); 99.66 ($-\text{CO}-\text{O}-\text{CH}-$); 122.30-143.20 (C- arom. rings); 165.18 (C=O amide); 170.41 (C=O oxazepin ring); 179.80 (C=S)
15	21.4 (CH_3); 55.6 (OCH ₃); 122.45 ($-\text{CO}-\text{O}-\text{CH}-$); 123.36-145.44 (C- aromatic rings); 166.90 (C=O amide); 175.32 (C=O oxazepin ring); 175.55 (C=S)

Examination of antibacterial activity (24):

In order to perform the antibacterial activity of some of the prepared compounds; we used the disc diffusion technique by using two types of bacteria: *Staphylococcus Aureus* and *Bacillus* (gram-positive bacteria) and *Escherichia Coli* and *Pseudomonas* (gram-negative bacteria). A sterilized filter paper (Whatman no.1, 5mm) disk was uploaded with 800 µg of the prepared compounds in each disk and placed on agar plates which contain (100 µL) of the bacteria under testing, then incubated for 1 hour at (5 °C) in order to get fine dispersion. After that, further incubation was made for 24 hours at (37 °C). The zone of bacteria growth inhibition was determined.

As shown in Table (6), some of the prepared compounds show different biological activities that ranged from strong to weak against specific type of bacteria, while some have no effect. The compound 3 shows strong inhibition of *S. Aureus*, while the compounds 2 and 9 strongly inhibit the growth of *E. Coli*. The compounds (8 and 11) strongly inhibit *Bacillus* growth. A weak inhibition activity of *Bacillus* bacteria was found by compounds (2, 3 and 15)

while compound 13 shows no inhibition activity on *Bacillus* bacteria growth. The compounds (8, 11, and 13) show modest inhibition activity on *Pseudomonas*, however, compounds (2 and 3) show no affect on *Pseudomonas* growth.

Table 6: Antibacterial activity of select compounds

Compound No.	Staphylococcus Aureus +ve	Bacillus +ve	Pseudomonas -ve	Escherichia Coil. -ve
2	7	5	-	12
3	11	5	-	9
4	8	7	12	-
8	5	11	8	-
9	8	9	4	12
11	3	13	9	4
13	-	-	7	8
15	-	4	11	-

DMSO (Solvent) [C]: 800µg/ml, Zone of inhibition

- 1- (11-15) strong
- 2- (9-10) moderate
- 3- (3-6) weak
- 4- (-) no inhibition

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